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# THE METALLOGRAPHIST

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A Quarterly Publication devoted to the Study of Metals, with  
Special Reference to their Physics and Microstructure,  
their Industrial Treatment and Applications

EDITED BY  
ALBERT SAUVEUR

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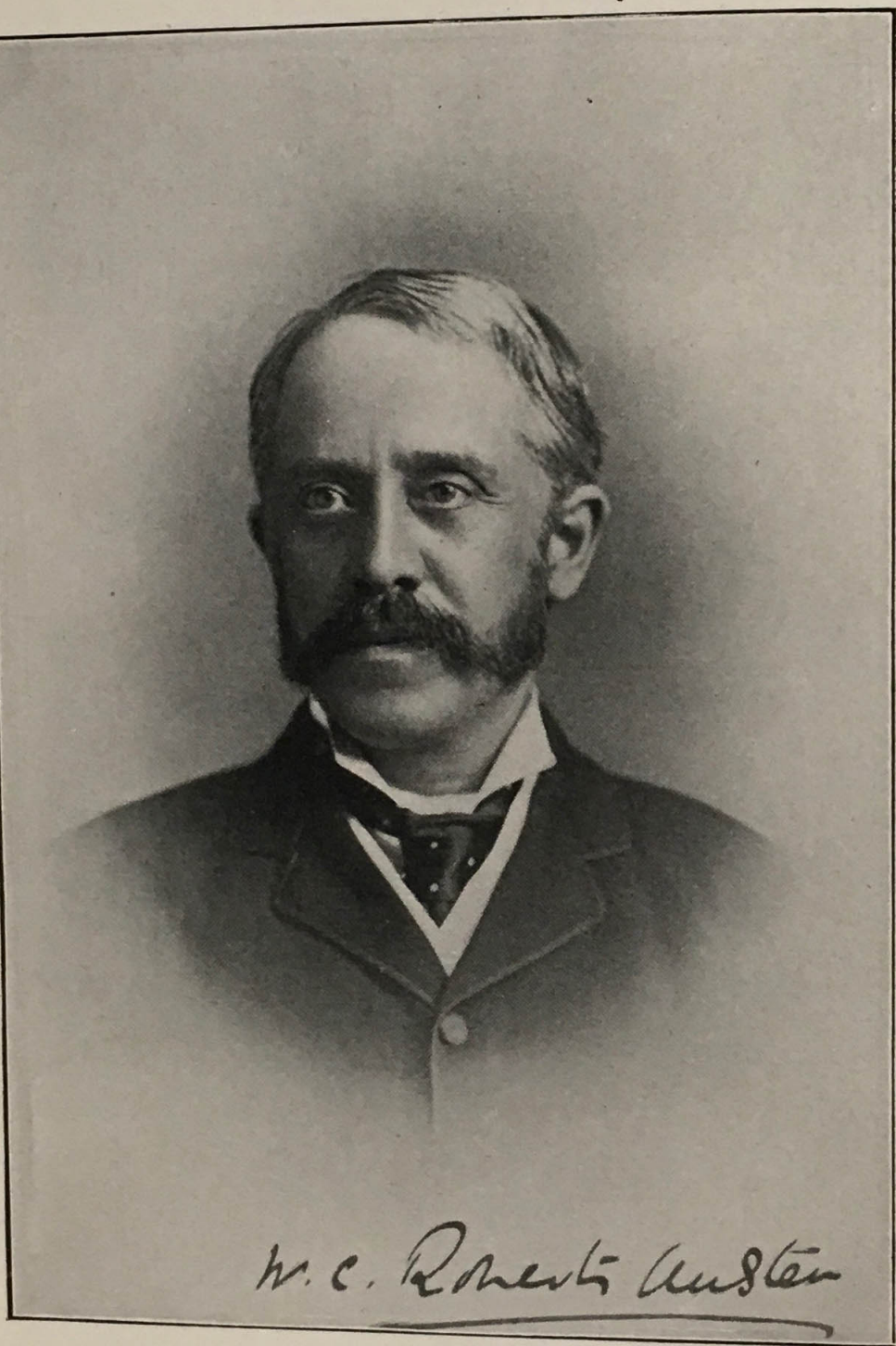
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# The Metallographist

A QUARTERLY PUBLICATION DEVOTED TO THE STUDY OF METALS, WITH  
SPECIAL REFERENCE TO THEIR PHYSICS AND MICROSTRUCTURE,  
THEIR INDUSTRIAL TREATMENT AND APPLICATIONS.

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Vol. VI

JANUARY 1903

No. 1

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## SIR WILLIAM ROBERTS-AUSTEN

BY the death of Sir William Roberts-Austen metallurgy loses one of its most brilliant ornaments, a natural leader of thought, a fascinating teacher, an indefatigable and talented investigator, and a personality of rare attractiveness.

Born in 1843, he entered the Royal School of Mines in 1861, and, shortly after his graduation, he began at the Royal Mint that career which has been so brilliant and useful. At the age of twenty-six he was appointed assayer of the mint, and at thirty-nine he assumed all the responsibilities of the post of assay-master. Here he remained up to the time of his death, performing arduous duties and carrying a weight of responsibility which would have been quite burden enough for a man of any usual strength. It is within the present year, if my memory serves me right, that he was appointed Deputy Master of the Mint. He was probably by far the most important authority in the world on the technique of coinage.

In 1880 he succeeded the illustrious Dr. John Percy as Professor of Metallurgy at the Royal School of Mines, carrying on the duties of this office as well as those of assayer of the mint. This chair, too, he occupied throughout the rest of his life.

Besides these two very responsible and difficult posts, he was active in the work of many scientific societies, and in late years gave his time and strength without stint to the work of the famous Alloys Research Committee of the Institution of Me-



chanical Engineers, and also to that of many public committees and royal commissions. It is no secret that he intended to resign his post at the mint during the coming year, for the burdens which he had been carrying lately were too great even for his wonderful powers.

Honors naturally were showered upon him. He was an honorary member of the American Institute of Mining Engineers, of the British Institutions of Civil Engineers and of Mechanical Engineers; a Knight of the Legion of Honor of France; a K.C.B.; President of the Iron and Steel Institute, and a fellow of the Royal Society at the age of thirty-two.

Such a man leaves no monument commensurate with his powers and his accomplishments. He was too busy serving his fellow-men to have time to raise a monument to himself. His most important works are the five reports of the Alloys Research Committee, and his "Introduction to the Study of Metallurgy." These are works of great merit; but important as they are, they do not begin to represent his labors; they do not even suggest them. He may almost be said to have been the Alloys Research Committee, so great is the proportion of its work which is due to him. Besides these he wrote very many scientific papers, of which the earlier ones were under his former name of W. Chandler Roberts.

As a lecture-room demonstrator he had remarkable gifts. His experiments were brilliant, and his explanations very lucid and fascinating.

But over and above all these symptoms of his powers, we must look at the beautiful character of the man. It was not what he did, though this was wonderful enough, but what he was. Here was a gentleman and a scholar; a man not only of originality, initiative and action, but of sweetness and light; of high and rare gifts, but better still, of high and beautiful character. But character must be felt; it is not susceptible of mathematical demonstration.

To his original and courageous mind it seemed that he could serve science best by applying himself, not as his fellow chemists in general did, to pure science, to the common aqueous methods of the usual chemical laboratory or to organic chemistry, but to that very difficult applied science, metallurgy. He strove to interpret and to reduce to order the perplexing phenomena of his



art, knowing that, in the eyes of his British fellow-men of science he was thereby raising a prejudice against himself. That such a prejudice against applied science was very strong in the days when he cut out that path of usefulness from which he never swerved, is familiar to all whose memory runs back even half way to that period. But for the sake of science, which is merely another way of saying for the sake of serving his fellow-men in the way for which he was fitted, he did not shrink from undergoing this prejudice. Bringing to metallurgy as he did a vast store of precious knowledge, of knowledge all the more precious because it was of a different class from that with which other metallurgists were equipped, he approached the subject from a standpoint different from that of others. It was well that he did.

Most admirable, and most elevating as an example, was his devotion to his work, although he had every temptation to lay it aside for a life of ease, in which he could have gratified his strong æsthetic tastes. But he deliberately chose to labor as few can do, at the cost of great physical suffering, and of inevitably shortening his life.

Here was one who loved his fellows, and for them deliberately shortened his life and suffered, disdaining the life of ease which was open to him.

HENRY MARION HOWE.

*Columbia University in the City of New York,*  
December, 1902

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## EFFECT OF SUPERHEATED STEAM UPON THE TENSILE STRENGTH OF ALLOYS\*

By J. L. HALL

THE use of superheated steam of high temperatures has forced the engine builder to take cognizance of certain questions which have heretofore needed no particular consideration. Not the least important of these is the effect of such high temperatures on the physical properties of sundry materials of construction.

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\* Received October 9, 1902.



Can we expect the same length of life from such materials in contact with highly superheated steam, as with saturated steam of the same pressure; or, will the structure, and consequently the life of the materials, be adversely affected by changes in the physical condition of the working fluid; and will such adverse effect, if any, be produced immediately; or, is time an important element in effecting the change?

A few months since the writer had occasion to investigate this matter; and it was in attempting to find data pertaining thereto that the lack of such information was forcibly impressed upon his mind. To establish the non-existent data, a series of experiments was carried out, as outlined in the following notes, which are written with the hope that the description of the methods employed, and the results obtained, will be of benefit to others seeking information on the same subject, and that they will also provoke an interchange of ideas mutually beneficial to all.

This particular investigation was confined to the effects on two different alloys of copper, as experience has quite generally indicated that copper and some of its alloys have proven unreliable when subjected to the action of highly superheated steam.

It has been suggested that this deterioration might be due to the repeated heating and slow cooling as the steam was turned on and off the apparatus in which this material was used, as the slow cooling being diametrically opposed to the usual annealing process for copper, might cause it to become brittle. This, of course, would imply that time was an important element in producing this effect, or more strictly speaking, that the effect of numerous repetitions of the process was cumulative. The first set of experiments was undertaken to determine the truth of this statement, as applied to a special bronze. Specimens about five inches long, cut from a rolled rod  $\frac{3}{8}$  inch in diameter were heated to  $320^{\circ}$  C. in an electrical furnace, and cooled slowly in the air, reheated and recooled, until the process had been repeated twenty-five times. A second set of bars was then treated to one heating and cooling, the same maximum temperature being attained as with the first set. A third set was heated to  $440^{\circ}$  C. once, and slowly cooled in the air, and a fourth set was reserved untreated for the purpose of comparison with the other three.

The temperature of  $320^{\circ}$  C. was selected because we had been informed that the bronze would show decided weakness after



heating to this point, and because this represented the probable maximum limit to which the alloy would be subjected in practice.

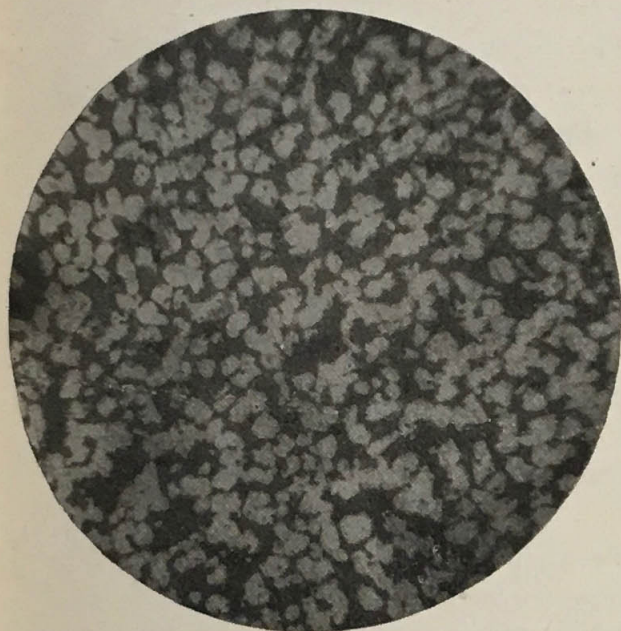


Fig. 1. Heated to 320° C. twenty-five times and cooled in air.

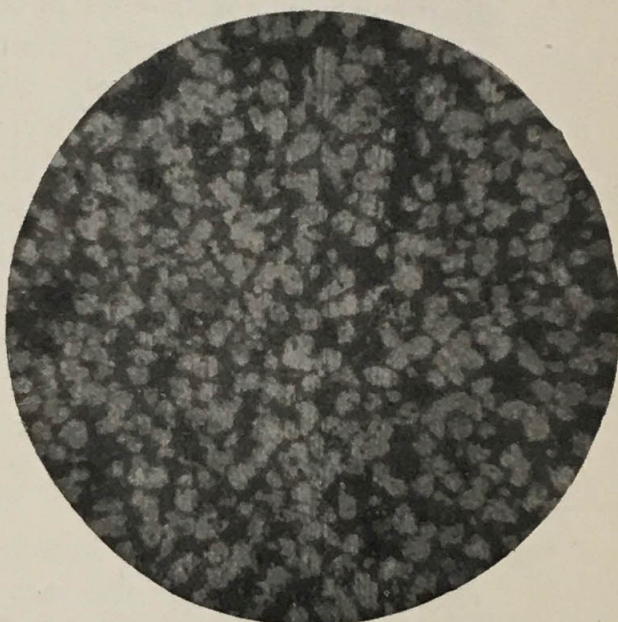


Fig. 2. Heated to 320° C. once and cooled in air.

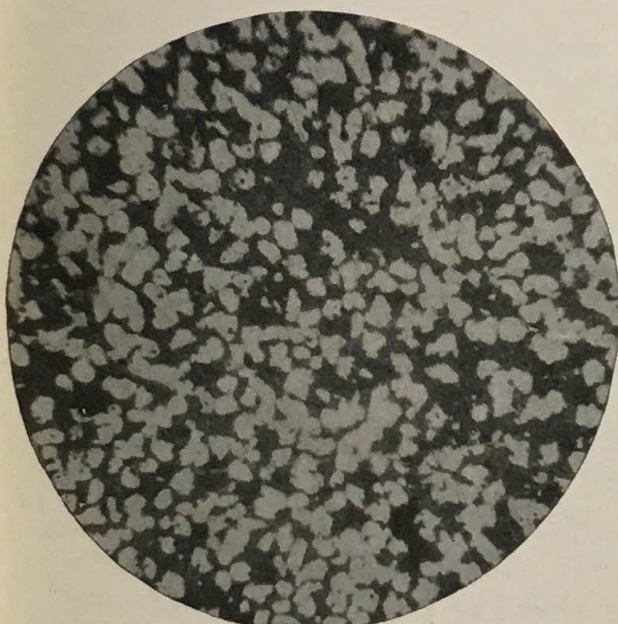


Fig. 3. Heated to 440° C.

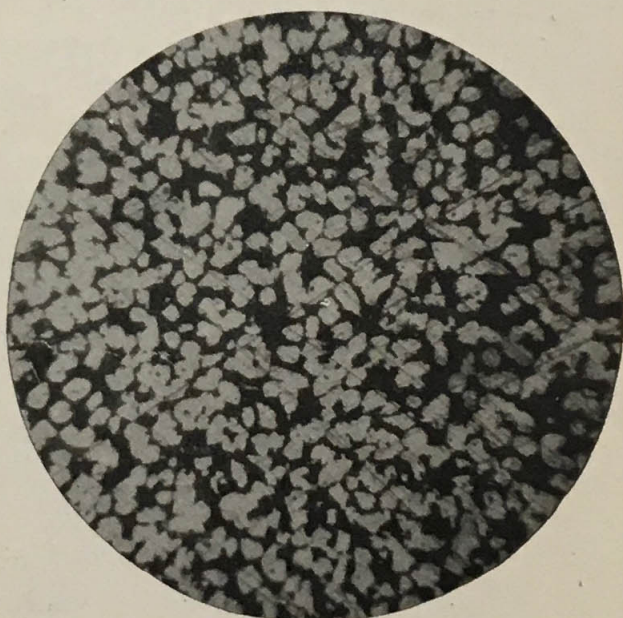


Fig. 4. Untreated sample.

Wishing, however, to observe the effect of heating to a somewhat higher temperature, the third set of bars was treated as mentioned in the preceding paragraph.



After subjecting the specimens to the heat treatment, a section was cut from each, polished and etched, and examined under the microscope for changes in structure. Fig. 1 shows the structure when heated to  $320^{\circ}$  C. twenty-five times, and cooled in the air; Fig. 2 when heated only once; Fig. 3 when heated to  $440^{\circ}$  C.; and Fig. 4 the structure of the untreated specimen. All the photographs show a magnification of 95 diameters. The microstructure indicates an alloy containing approximately 40 per cent zinc, made up of white rounded crystalline grains in a dark amorphous magma, the size of the grains depending upon the rate of cooling and the temperature to which the alloy was heated. The finer the grain, the greater the strength of the alloy. We assumed that the lighter portions were more malleable, while the hardness of the material depended upon the ground mass or dark portions.

After removing the section for microscopic examination, the remaining portions of the bars were formed into test pieces and subjected to tensile tests in an Olsen Testing Machine with the following results:

No. of Test	Average Diameter of Test Pieces in Inches	Average Ultimate Tensile Strength in Lbs. per sq. Inch	Average Elongation	
			In Two Inches	Per Cent.
1	.295	76600	.58	29.0
2	.295	76090	.53	26.5
3	.295	75280	.60	30.0
4	.294	80480	.44	22.0

NOTE.— The test numbers in the above table refer to corresponding figures.

From a consideration of the above table it would appear that the tensile strength of the bronze was lessened after the first heating and cooling from  $320^{\circ}$  C., but that subsequent treatment of this nature had little effect upon the ultimate strength, although the elongation seemed to increase after the first treatment, and slightly decrease upon repetition of the same. With a higher maximum temperature of heating, the strength remained about as determined by the first set of experiments, while the elongation increased. These results seem to agree with the deductions made from the microscopical examination.



To determine the ultimate strength of the bronze while at the maximum temperature of  $320^{\circ}$ , we conducted a second series of experiments, the first step of which was to find the best method of keeping the test pieces at the temperature selected, for a period of time sufficient to make the tests for tenacity; the second to make the tensile tests and compare the results with the strength of the alloy when cold; and the third to repeat the process upon a second alloy from which we believed equally good results should be obtained. The first step indicated that we could, with the apparatus employed (which will be described later) maintain the specimens at temperatures ranging from  $330^{\circ}$  to  $400^{\circ}$  C. for an indefinite period of time. The second step gave results made up of averages of several tests as follows: The first alloy, showing an ultimate strength per square inch when cold of 85,160 pounds, gave, when at the temperature of  $320^{\circ}$  C., but 33,735 pounds, or a reduction of 60.4 per cent in strength; and the second alloy, under similar conditions of treatment, gave a reduction in strength of 63.8 per cent, although this second alloy was stronger when cold than was the first. The difference in strength in the first bronze, as shown by the experiments recorded early in these notes, and its strength in the second set, is possibly due to the fact that the test specimens were from different rods, probably bought at different times, although they were supposed to be of the same composition.

The elongation of the specimens at  $320^{\circ}$  C. is apparent from an examination of Fig. 5, where numbers 1 and 5 are photographs of specimens of the two alloys, whose tenacity was determined when cold, and numbers 3 and 6 when at the higher temperature. The time occupied in drawing these two last mentioned specimens to the small area shown was quite appreciable compared with the time occupied in producing rupture when cold.

The apparatus for heating the test pieces consisted of a steel cup  $2\frac{3}{4}$  inches in diameter and 4 inches deep, from the bottom of which projected a stem  $1\frac{1}{2}$  inch in diameter and 4 inches long, fitting into the lower jaws of the testing machine, and having the end inside the cup drilled and tapped to receive the test piece; another stem similarly drilled and tapped, screwed over the other end of the test piece and fitted into the upper jaws of the machine. Filling the cup with a molten lead bath, and placing the apparatus in the testing machine, the tensile strength of the specimen could



be determined while the metal was at the proper temperature. Heat was applied to the outside of the cup to maintain this temperature. When using this apparatus with low degrees of heat, a frame wound with wire can be slipped into the cup, which, in this case, is filled with oil having a high flashing point, and by connecting the coils to a source of electricity, the oil can be readily heated.

In conclusion, to complete the investigation, it would have

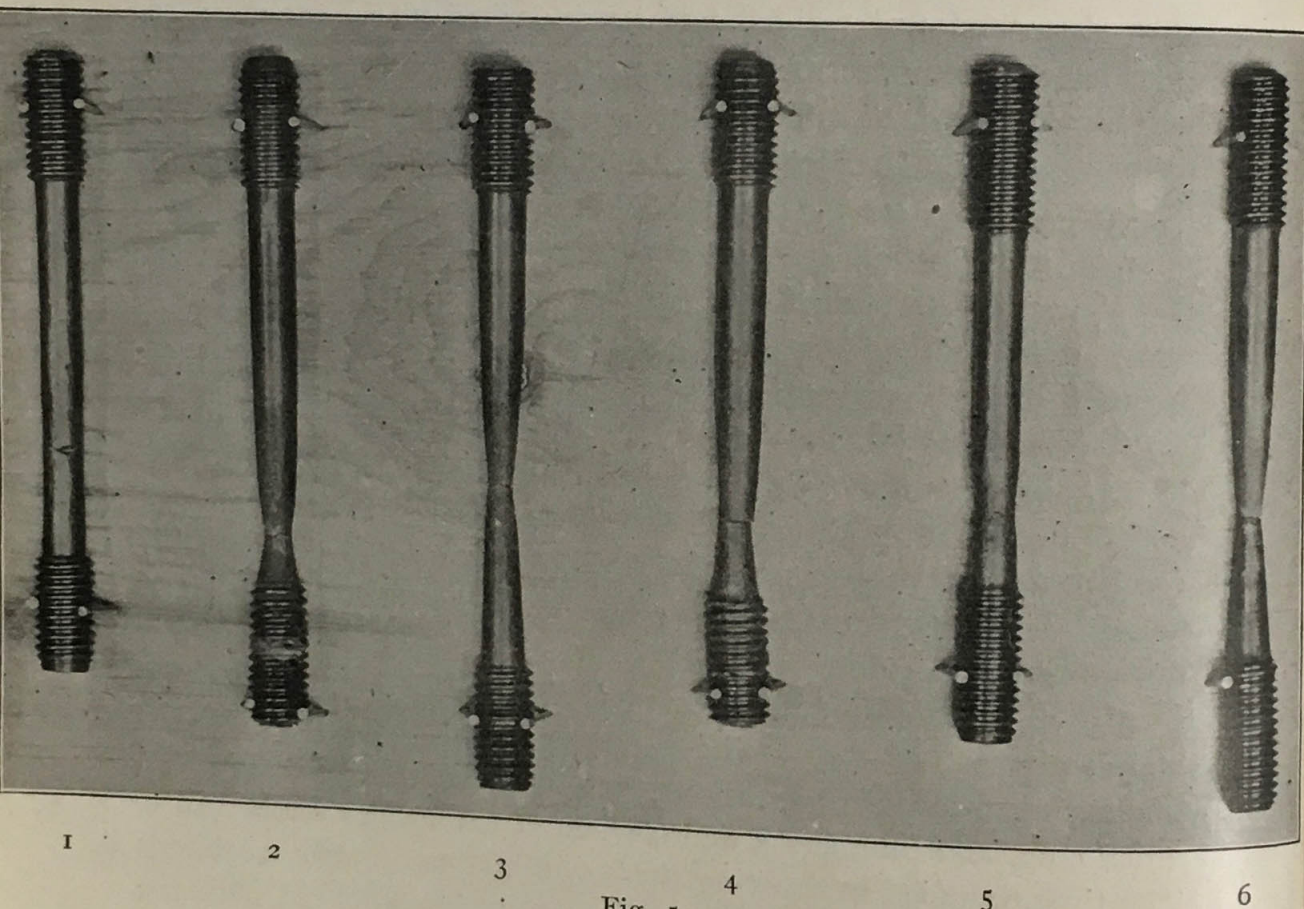


Fig. 5.

been well to have made careful chemical analyses of the bronzes, together with other sets of tests under different degrees of temperature, but as intimated earlier in this paper our object was to determine the effect of a certain set of conditions upon a certain selected alloy, representing commercial practice, and it was in no way a laboratory investigation.

It is our hope to continue such tests on other alloys, with the object of securing data enabling us to make an intelligent selection of the metal best suited for given conditions.



# ON THE SIMULTANEOUS PRESENCE OF FERRITE AND CEMENTITE IN STEEL \*

By E. F. LANGE

IN a paper entitled, "The Microstructure of Steel and the Current Theories of Hardening," presented to the American Institute of Mining Engineers at the Colorado meeting, September, 1896, Mr. Albert Sauveur describes the microscopic constituents of steel. Referring to the constituent cementite he says:

"This is iron combined with cement-carbon, i.e., carbon as it exists in unhardened steel. It is a carbide answering to the formula  $Fe_3C$ . . . . Like ferrite, cementite occurs:

"(1) In segregated masses in very hard steel, but it always remains structureless. . . .

"(2) As a very thin membrane, around the grains of pearlite when present in small quantities and under certain conditions.

"(3) As a structural element of pearlite. . . . *Structurally free ferrite and structurally free cementite never exist together in the same steel.*" The italics are mine.

The object of this note is to criticize this last statement, which appears to be far from accurate in the light of recent researches.

As a matter of fact we repeatedly obtain, in our own practice, annealed steel castings (of the approximate composition Comb. C .38, Si .25, Mn .70, S .03, P .05) which exhibit a microscopic structure consisting entirely of structurally free cementite and structurally free ferrite. A notable fact is that some of the very best tests we have obtained have been made on pieces having such a micro-structure. A steel of this character gave the following tests, which prove the exceptionally good quality of the material.

Ultimate stress	. . . . .	29.60 tons per sq. inch.
Elongation % on 2 inches	. . . . .	38.0
Reduction of area %	. . . . .	55.0
Proportion of elastic limit to ultimate stress		51.2
Combined Carbon	. . . . .	.38
Bending angle on a bar of 1 sq. inch cross section		180° unbroken.

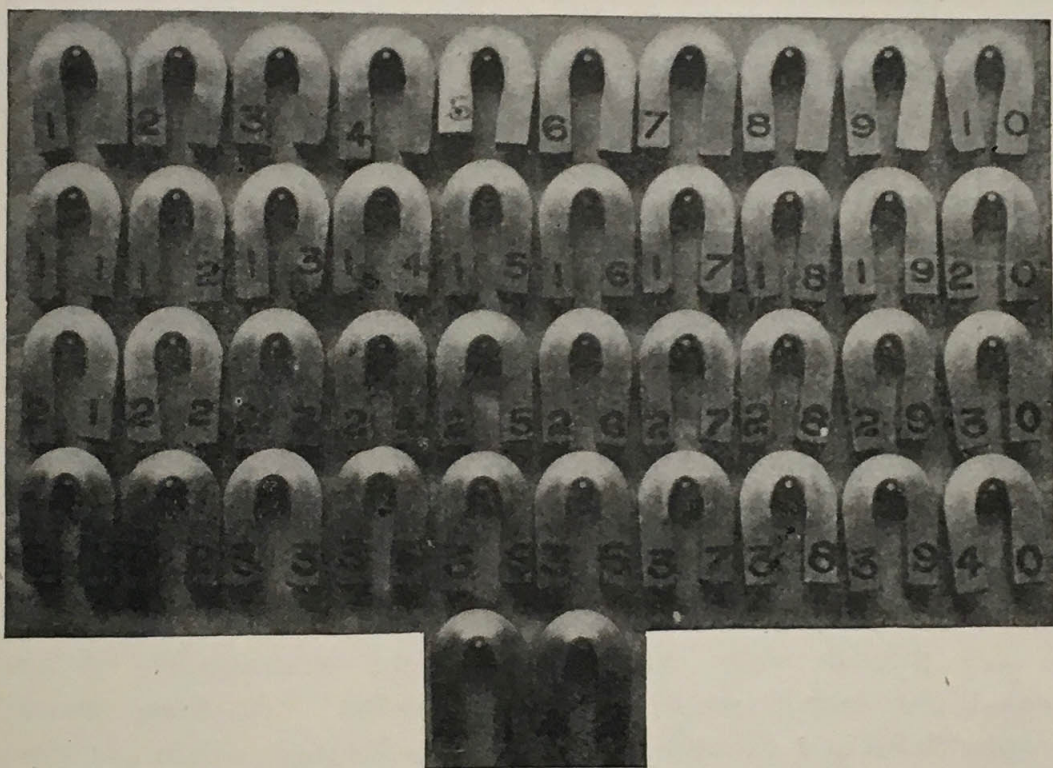


The structure referred to contains absolutely no pearlite. It consists of ferrite crystals, throughout which little balls of segregated cementite are scattered. These balls or globules are generally grouped together more or less, and evidently occupy areas where pearlite has been. Each globule is, however, entirely separated from its fellows, and is completely surrounded by ferrite. In other words, the pearlite has been completely resolved into structurally free cementite and ferrite. The globules of cementite have a tendency to group themselves in the corners and along the junction lines of ferrite crystals; precisely, in fact, as the pearlite from which they have segregated, would have been arranged. The cementite is easily distinguishable from the ferrite by its brighter appearance. As an additional confirmation of the fact that the globules really are cementite, a section was polished and heat-tinted to blue (of course without etching) when the cementite was plainly distinguishable as dots of a blood-red color against a blue background of ferrite. The complete segregation of cementite is not observable in all our annealed castings by any means, though evidences of incipient segregation are always very marked. In many sections the pearlite areas contain globules of free cementite in their midst surrounded by a dark granular mass of unsegregated pearlite. In cases where the segregation of cementite is at all far advanced, laminated pearlite is seldom visible in the section. A very slight difference of annealing temperature is apparently necessary to produce a considerable modification in the structure. We often obtain, from the same annealing, steels in which the cementite segregation is complete, and others in which the segregation is not nearly so far advanced. Size of grain affords no indication as to the character of the pearlitic areas. Some microsections of annealed steel castings with completely resolved pearlite were sent to Mr. J. E. Stead for inspection. He replied: "... I have examined the two microsections you have sent, and note that the cementite of the pearlite is more or less segregated. There is no doubt at all about the fact that cementite and ferrite may be structurally free in the same steel. I have frequently met with cases in very soft steel in which there has been no pearlite, the cementite having segregated in little masses. Osmond, I think, was the first to notice this peculiarity. A photograph I have shown in my paper on "The Crystalline Structure of Iron" illustrates this. We now know that the segregation



takes place at a very few degrees below  $Ar_1$ , and in all probability you do obtain it on very slow cooling between  $700^\circ$  and  $600^\circ$  . . .”.

This last remark of Mr. Stead's is doubtless quite true; but in our case it appears to be the maintaining at a particular temperature for the long period of time that determines the segregation of cementite since the rate of cooling from  $700^\circ$  to  $600^\circ$  in our case is not excessively slow. The steel Mr. Stead refers to in his "Crystalline Structure of Iron" paper is a Swedish Bessemer steel of the composition Comb. C .04, Si .05, Mn .27,



S + P .03. It had excessively coarse granulation and was returned to the steel-maker as rotten. Some of the grains measured  $\frac{1}{4}$ " across the face. The carbon in this section was entirely in the form of "massive cementite laid between the larger grains at their junctions." Mr. Stead is satisfied that this was rendered brittle by heating for a long time in either an annealing or ordinary heating furnace at a low temperature. *The segregated cementite affords proof that it was very slowly cooled between  $700^\circ$  and  $600^\circ$ .*

In his correspondence on Arnold and McWilliam's paper on the "Microstructure of Hardened Steel" (*Journal Iron and Steel Inst.* Vol. I. 1902, p. 172) Mr. Stead refers to some ex-



periments he made of heating several small bars of steel for several hours at one end only to a temperature of  $800^{\circ}$  or  $900^{\circ}$  C.; the heat being graduated from that temperature at one end to far below redness at the other. The bars were sectioned along their length after cooling. Mr. Stead observed that in a range between  $620^{\circ}$  and  $670^{\circ}$  C. there was a very marked segregation of the ferrite and cementite of the pearlite.

Several other observers have noticed structurally free ferrite and cementite in the same steel, though we have yet to learn that the structure is obtained in regular practice by any other steel makers or users.

In Professor Arnold's paper on "The Properties of Steel Castings" (*The Metallographist*, Vol. V, pp. 9 and 10), he mentions that in several of his annealed sections the pearlite is "badly defined, owing to segregation of the carbide striæ into massive cementite." He, however, obtains nothing like the complete segregation that we have obtained. Professor Arnold's process of annealing is to maintain at  $950^{\circ}$  C. for 70 hours and then cool slowly, — the castings being cool enough for drawing in another 100 hours. The time occupied in passing from  $700^{\circ}$  to  $600^{\circ}$  on cooling is about 6 hours.

Henry Fay and Stephen Badlam in a paper on "The Effect of Annealing upon the Physical Properties and the Microstructure of a Low Carbon Steel" (*The Metallographist*, Vol. IV, p. 31) publish some nondescript-looking photographs of annealed low carbon steel "polished in relief." In these photographs there is a ground mass of ferrite through which particles of another constituent are disseminated, which the authors "would expect to be pearlite, but which looks very much more like structurally free cementite." But if the authors, who have seen the actual sections are not quite sure of the nature of this constituent, it is hardly possible for outsiders to form an opinion from the photographs.

To sum up: There can be no possible doubt, as Mr. Stead says, that structurally free cementite and ferrite may be obtained in the same steel. The conditions favorable to the formation of this structure are an extremely slow cooling between  $700$  and  $600^{\circ}$ .

The photograph reproduced here illustrates the great increase of ductility which we obtained by our method of annealing castings.



### Postscript

BY ALBERT SAUVEUR

Mr. Lange appears to have removed all doubt as to the possible presence of structurally free cementite and of structurally free ferrite in the same steel. When the writer in 1896 gave it as his opinion that the co-existence of these two constituents was not possible, he had never observed it, and to the best of his knowledge it had never been alluded to by other observers. On theoretical grounds his statement was fully justified, for when the two components of a binary alloy form, as is the case with steel, a eutectic alloy, they are never found together in the same sample. Structurally free silver and structurally free copper cannot, to the best of our knowledge, exist together in any silver-copper alloy. Such alloys always consist of a certain amount of the eutectic mixture, the balance being made up of one or of the other constituent. If we are right in considering steel as an alloy of iron and carbon forming a eutectic mixture (pearlite), then the simultaneous occurrence of the two components in the same sample is decidedly abnormal and remains unexplained.

It is not surprising that such a metal should possess a high ductility, for the small particles of cementite disseminated throughout the mass of soft ferrite could not greatly decrease the ductility of the metal as a whole.

## SOME UNUSUAL CRYSTALS OF CAST IRON \*

By J. E. JOHNSON, Jr.

THE illustration herewith shows some masses of crystals which are perhaps interesting in themselves to those interested in the study of cast iron, since most people seldom or never see crystals of that material of such size; but more interesting in view of their origin and their analysis, considered together. The analysis is as follows:

Total Carbon . . . . .	6.68
Graphitic Carbon . . . . .	6.08
Combined Carbon . . . . .	0.60
Silicon . . . . .	.47
Sulphur . . . . .	.220
Manganese . . . . .	0.89
Phosphorus . . . . .	0.83

These pieces of iron were taken from the outer edge of the hearth or bottom of a blast-furnace which had been running for about three years, making, practically all the time, iron for the basic open-hearth process, which ran about as follows:

Silicon . . . . .	.70
Sulphur . . . . .	.030
Phosphorus . . . . .	1.00
Total Carbon . . . . .	3.60
Manganese . . . . .	1.00

As all furnacemen know, the iron gradually eats up the hearth and lower part of the crucible of a furnace, and substitutes therefor a heterogeneous mixture of iron, lime, coke, altered fire-brick, etc., the inner portion of the mass being nearly or quite all iron, while the outer shades off gradually into the original fire-brick; as the heat of the crucible is higher or lower this bottom "takes up" or goes down; but of course the outermost portions of this mass are only actually molten during the

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\* Received in July, 1902.



hottest periods of the life of the bottom, and it is perfectly conceivable that iron might eat its way out to a certain position quite early in the blast and then be chilled there by the outside cooling of the crucible, never to be melted again throughout the length of the blast, although constantly almost in contact with the molten iron in the hearth, and in fact to a greater or less degree this is actually what happens, though of course the proportion of the whole length of the blast through which the iron remains there, may be much less than that stated; but there is ample reason to be sure that there is formed such an outside layer, which, with regular furnace work, remains for months in a practically constant condition just below the melting point.

It will be seen that these conditions account in very large degree for the composition of the mass under consideration, which evidently began by being approximately the normal iron produced in this furnace, and while in the bottom or outer edge of the molten iron in the hearth was entrapped in the solid portion of the "bottom," presumably when the furnace at some time got colder; the bottom "took up," never more to be remelted to the same depth during that blast, and the iron constituting this specimen simply lay there, perhaps molten itself; but more probably in a pasty condition surrounded by pieces of coke which were impounded with it and perhaps in contact on top with the base of that column of incandescent coke, which, whatever may be the case in other furnaces, undoubtedly stands on the bottom in this one. Thus separated from the desulphurizing influence of the slag by the supernatant liquid iron, our specimen simply proceeded to soak up sulphur and carbon from this coke; being assisted probably as to the former by the fact of its comparatively low temperature, at which the absorption of sulphur is undoubtedly facilitated. As to the latter element, the saturation point of iron for carbon being somewhere about 4.5 per cent, and the oxidizing influence of the blast or the access of any possible piece of unreduced ore or other oxidizing substance being cut off by the liquid iron and slag above it is perhaps not surprising (in view of the indefinitely long time of contact of the specimen with incandescent coke) that the carbon content should be as high as it is.

It was from this zone, after the furnace had been blown out and the bottom had had a week or two in which to cool, during



repairs to other parts, that the specimens in the photograph were taken, and while the photograph is a beautiful one in its clearness, I am sure Mr. Sauveur (who was kind enough to take it) will agree with me in saying that it does not fully give a sufficient idea of the size of the crystals, though it does show, to any one who takes the trouble to examine it carefully, the way in which the whole mass is made up of them alone, without practically any other kind of structure.

As for size, the largest piece shown is about the size of one's fist, and many of the larger crystals measure nearly or quite an inch across.

The faces of these are covered with real plates of graphite which adhere very firmly to them, so much so as to make one wonder whether it could be anything else; but the color and lustre are so characteristic as to leave little doubt of the fact.

The shapes of the crystals may be seen quite clearly from the photograph, the angles being very sharp in many cases; but whether this is caused by the accidental meeting, at these angles, of a series of flat flake-crystals, or whether the faces meet with definite solid angles and represent true crystals, simply confused by the way in which they are aggregated, the writer hardly feels competent to say.

The notable thing about these masses next to their unusual appearance is their high content of total carbon and of sulphur, and the high proportion of graphitic carbon with this proportion of sulphur, which leads to what is the really notable circumstance of the whole thing; that such a structure should obtain with such a content of sulphur. The scientific value of this lies in the strong proof it constitutes of the fact that as far as their effect on the granular structure of cast iron is concerned, slow cooling and sulphur are precise antitheses of each other.

There are many illustrations which might be given of this fact; but one of the most interesting is that supplied by some plates of iron which had been built into the upper part of the lining of the same furnace to protect the lining from injury by the impingement of the stock and which were partly melted by the high heat in the upper part of the furnace when blowing out.

Some of these, broken during repairs, were seen to glisten like silver and with a totally different lustre from that of cast iron, so much so as to excite great interest as to what they could



be, before they were actually reached. They were then found to be these plates which had been heated so hot on their exposed faces that they had partly melted and the center had run out. The top and bottom being kept cooler by the contact of the brick work had not melted and the thin plates of metal remaining, say  $\frac{3}{8}$ " thick, so altered that they showed, on fracture, crystals sometimes a quarter of an inch across and of a true metallic lustre like silver as mentioned above. On analysis these showed:

Sulphur	.	.	.	.	.	.	.124
Phosphorus	.	.	.	.	.	.	.80
Silicon	.	.	.	.	.	.	0.11
Manganese	.	.	.	.	.	.	0.26



Having been cast originally from iron not very different in composition from that of the standard basic, quoted above, it seems to be fair to assume that these absorbed sulphur from the coke a little at a time during the blast, and that the carbon was oxidized out by the flame which melted the plate, the "blow-out" having been made with the bell out and the top open. The almost total absence of graphite was most noticeable in the color and nature of the crystals.

It is perfectly obvious to any one with a knowledge of cast iron that such a fracture would never be found with such an



analysis if the cooling had taken place at any ordinary rate, but protected and kept very hot, as the plates were, by the brick work, the large crystals of relatively pure iron, free from graphite, had a chance to form.

Perhaps the most striking illustration of the fact that sulphur and slow cooling have opposite effects is furnished by the small chill-cast samples taken from every cast at many furnaces, the small pig being about two inches wide on the bottom and an inch and a half thick with sufficient draft to bring it out of the chill-box in which it is cast.

With iron of less than one per cent silicon there is nearly always some chill, and with silicon at about 5 per cent or under, the whole sample is chilled clear through; but there is the greatest difference in the broken face when thus chilled solid, depending on the quantity of sulphur present. If the sulphur is low, say .025 per cent or under, the fracture will be markedly crystalline, with long prismatic crystal standing perpendicular to the bottom and each of the sides, and showing a clear line between the different sets of crystals, running from the center to the two bottom corners, the fracture being in one plane, normal to the axis of the pig, while if the sulphur is high, say .060 or over, the fracture will be conchoidal, with absolutely no crystals present, and frequently not even approximately in one plane; the intermediate percentages of sulphur corresponding to the graduations between these two kinds of fracture, so that a person familiar with a certain iron can judge very accurately of its sulphur contents by the fracture of the chill sample alone; *if the sample is always allowed to cool down to a black heat in the chill-box before quenching*; but if the sample of an iron of low sulphur content be taken out of the chill while still bright red and immediately quenched, the fracture will be precisely that of a high sulphur iron.

These are all facts of minor interest even from a scientific point of view; but the writer holds that it is only by the knowledge and grouping of a great quantity of these facts, in themselves almost insignificant, that thorough and satisfactory working knowledge of that complex substance, cast iron, will be reached, and the increased study put on this branch of the subject the past two or three years by competent metallurgists should serve as an inducement to all to contribute the unusual

facts which come to their knowledge, to the common store of information, as a possible aid to an even more rapid advance in this field.

### Postscript \*

BY HENRY M. HOWE

It appears to me that these are not crystals of iron properly so called, but that the iron simply cleaves along the plates of graphite, and that the form of the fragments is simply due to the shape of the graphite plates. At best we may call them pseudomorphs of iron after graphite. In the same way the magnificent crystals which we sometimes see in fractures of spiegeleisen are by no means of iron, but are rather the planes of cementite, and comparable with the sheets of mica which we see in the fractures of granite; just as granite very rich in mica will cleave along the sheets of mica so that mica is chiefly seen in the fracture, so these masses of iron break or cleave along the sheets of cementite or graphite, with the consequence that in pig iron, containing only perhaps 3 per cent of graphite, we see absolutely nothing but graphite in the fracture.

I have some very magnificent crystals of this kind in our museum, and I have for many years taken this view in my lectures.

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## SULPHIDE OF IRON: ITS PROPERTIES AND ITS CONDITIONS IN IRON †

By H. LE CHATELIER and M. ZIEGLER

THE harmful influence that small quantities of sulphur exert upon iron, and the remarkable action of manganese in counteracting this influence, have induced us to study more closely than has been done so far the relations of sulphur with iron and manganese.

*Crystallised Sulphides.* — A crystallised sulphide of iron is found abundantly in nature, in the shape of bi-sulphide or pyrites,  $\text{FeS}_2$ .

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\* Received, October 9, 1902.

† *Bulletin de la Société d'Encouragement*, September, 1902.



Under the influence of heat this substance loses more than half of its sulphur at a relatively low temperature, much inferior to that of the melting of iron, and there was, therefore, little probability of finding it in metallic products obtained by fusion. In fact, we have never found in metals any indication of its existence, and we shall not, therefore, consider it here.

A sulphide of iron is prepared artificially, which has a bronzed crystalline fracture, by melting some sulphur or some iron pyrites with metallic iron. It is generally considered to be a proto-sulphide of iron, and is used in laboratories for the preparation of sulphuretted hydrogen. Finally, the possibility has been indicated of obtaining in the laboratory some sulphides containing still less sulphur, but in reality, as will be shown by us, these so-called compounds are only mixtures, containing various amounts of metallic iron.

For our purpose, therefore, we need only consider the ordinary sulphide of iron, and it has seemed to us necessary to study it very closely before taking up the question of the relations of sulphur with iron, because *a priori* it seemed very probable that sulphur was present in that condition in steel.

*Chemical Composition.*—The analysis of this sulphide of iron, as it is found on the market, reveals a composition very different from that of the proto-sulphide. It is, however, quite constant, and contains about 0.8 equivalent of sulphur to one of iron. The analyses were made by the following method: The sulphide, finely pulverized, was digested for twenty-four hours with a mixture of equal parts of bromine and water. The residue was weighed, and the iron and the sulphuric acid present in the solution were determined. The results obtained are given below:

Iron . . . . .	53.2	64.0
Sulphur . . . . .	23.6	27.7
Insoluble . . . . .	24.0	7.2
Total . . . . .	100.7	98.9
Formula . . . . .	FeS <sub>0.8</sub>	FeS <sub>0.75</sub>

*Metallography.*—It was necessary in order to obtain an accurate knowledge of the constitution of this substance to study it by metallographic methods. This study offers some difficulty because the fragments of sulphide are full of cavities, and therefore difficult to polish even after filling the cavities with gum.



Three constituents are detected in a polished surface, before etching.

1. Some yellow grains constituting the bulk of the sample. They consist essentially of sulphide of iron.

2. Brilliant parts having a metallic lustre much more pronounced than the sulphide, and a white color very similar to that of iron. On being etched with a dilute solution of copper sulphate, this constituent is immediately covered by a deposit of copper; it evidently consists of metallic iron.

3. Finally, between the yellow grains, and frequently around the grains of iron, a substance is found made up of very fine

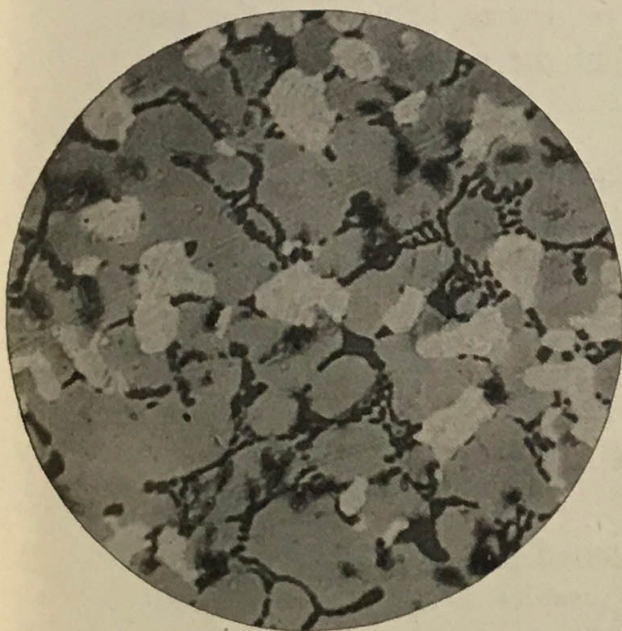


Fig. 1. Magnified 650 diameters.

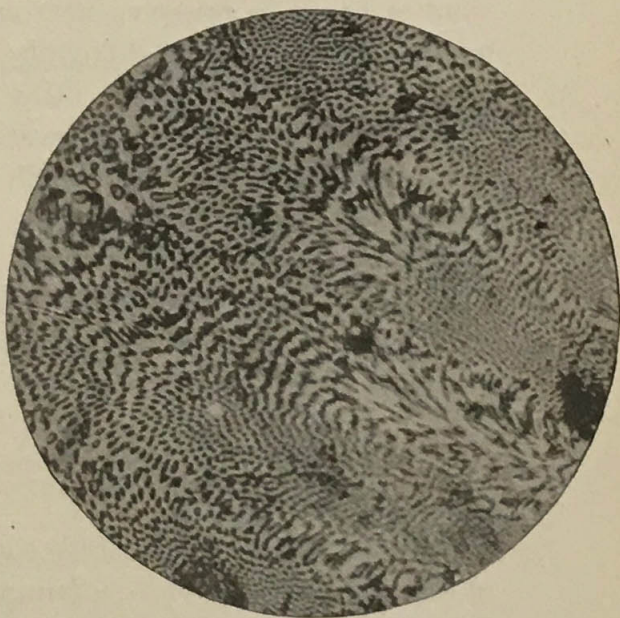


Fig. 3. Magnified 650 diameters.

plates having the characteristic appearance of eutectic alloys or of pearlite. One of the components of this eutectic is the yellow sulphide, while the other is a gray substance whose color recalls the appearance of the slag frequently found in iron and steel. We shall show later what is the constitution of this constituent.

Fig. 1 illustrates, under a magnification of 650 diameters, the appearance of sulphide of iron after polishing. The white areas indicate the iron, the gray meshes the sulphide, and the black spaces, the small cavities mentioned above. The pearlite may be seen between the gray grains.

Fig. 3, magnified 650 diameters, shows the structure of a sample containing a large amount of eutectic. It was selected



When the experiment is repeated several times, however, the contraction decreases at each operation, and finally the bar breaks in several pieces. The observed contraction, therefore, results from the reversible change of dimensions, the only interesting one to determine, and of permanent changes resulting from small cracks.

In order to ascertain with greater accuracy the temperature at which this transformation takes place, we have applied to this determination the dilatometer method. 132 grams of pulverized sulphur was introduced in a large thermometer having a capacity of fifty cubic centimeters, and the balance of the instrument filled with oil of vaseline. The thermometer was heated to the boiling point of the oil ( $300^{\circ}$ ), in order to expel the air completely, and it was then allowed to cool while observing carefully the temperature and the upper level of the liquid in the narrow tube.

The following results were obtained:

The contraction undergoes two disturbances, one between  $100^{\circ}$  and  $120^{\circ}$ , and the other above  $220^{\circ}$ . In reality there would be, therefore, two points of transformation, but the alteration of the oil above  $200^{\circ}$  has prevented a complete study of the second disturbance. Fig. 5 indicates the apparent increase of dilatation for every five degrees.

In order to ascertain more conclusively still the existence of this dimorphic transformation of the sulphide, we have studied the variation of electrical resistance, as the temperature varies. A bar of sulphide was cast, with two platinum wires inserted at both ends, by which an electric current was let through the substance, and between which difference of electro-motive force was measured. This bar was immersed in a bath of oil of vaseline and heated to  $330^{\circ}$ , with the following results: On heating, the electrical resistance increased at first regularly and considerably until at  $150^{\circ}$  it was twenty times its initial value. Above this temperature the resistance remained constant up to the highest temperature reached, that is,  $300^{\circ}$ . On cooling, the phenomena which occur are exactly similar, but of opposite direction, with only a slight retardation marking the changing point in the law of variation of the resistance. This point was lowered to  $130^{\circ}$ . It may be inferred that the precise point of the inferior transformation was located between  $130^{\circ}$  and  $150^{\circ}$ , or at a point  $140^{\circ}$ .



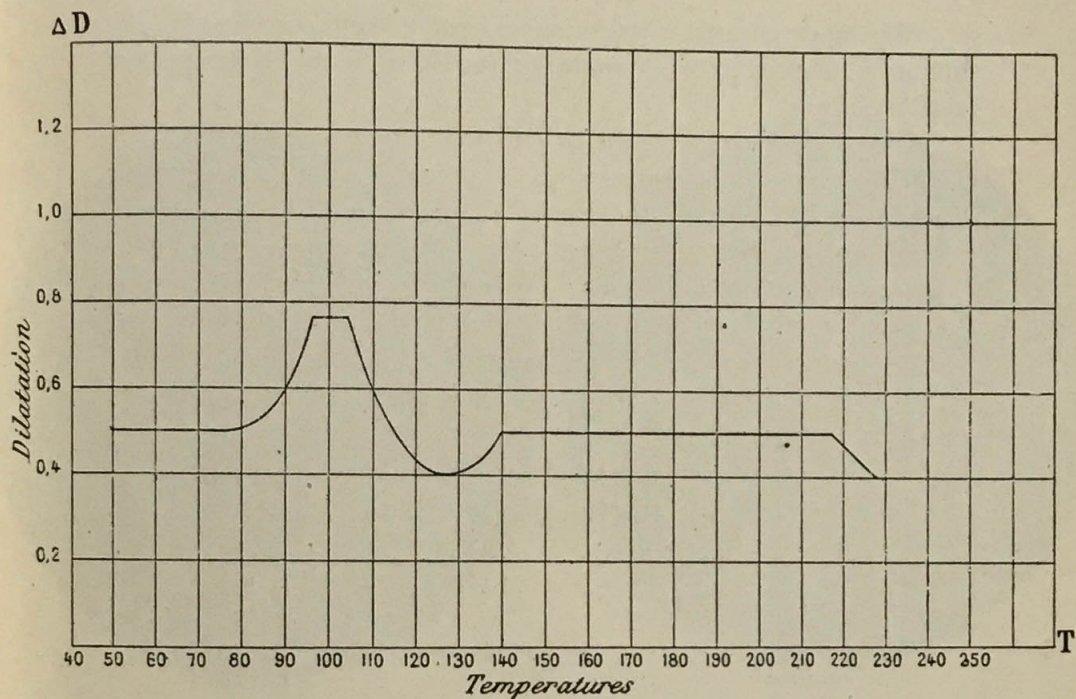


Fig. 5.

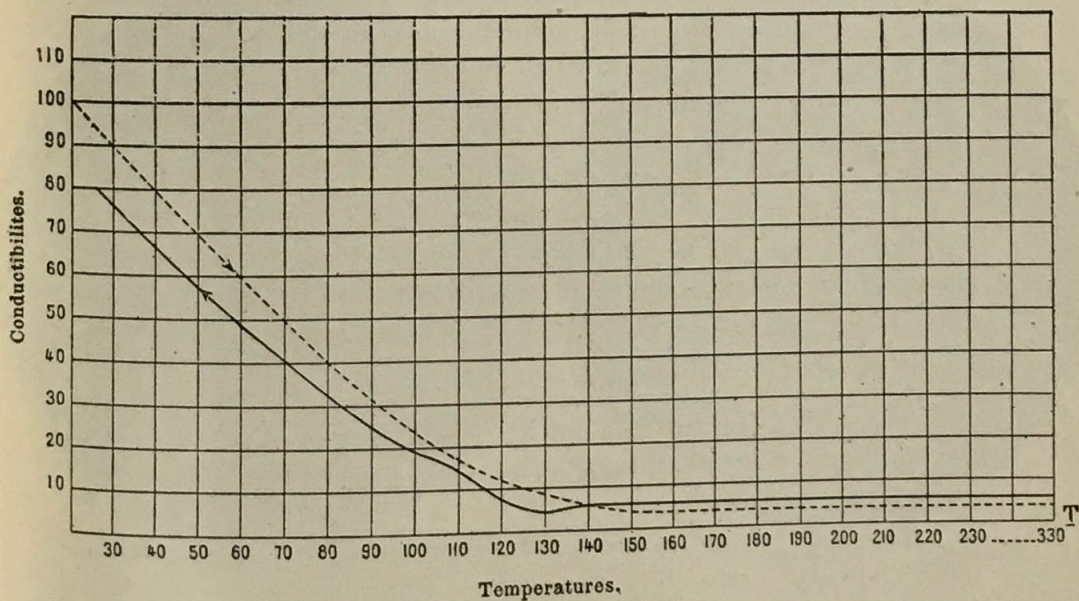


Fig. 6.

The upper transformation which had been revealed by the dilatation method did not manifest itself by a variation of electrical resistance.

Fig. 6 shows the results of this experiment.

The electrical resistances are expressed in functions of an arbitrary scale, because the irregularity of the bar made it impossible to determine the exact value of the specific resistance.

*Decomposition by Heat.*—When the sulphide of iron is heated to a high temperature it loses some of its sulphur, and the loss increases with the temperature. To perform this experiment iron pyrites were heated in a small porcelain crucible, which was covered and placed in another crucible filled with charcoal to prevent the access of air. The results obtained were as follows:

	Heated to 1200°	Heated to 1500°
Iron . . . . .	61.5	79.
Sulphur . . . . .	35.5	21.7
Insoluble Residue . . . . .	3.6	0.3
Total . . . . .	100.6	101.0
Formula . . . . .	FeS <sub>1.01</sub>	FeS <sub>0.48</sub>

The sulphide heated to 1200° corresponds very nearly to the proto-sulphide and is found, when examined under a microscope, to be made up exclusively of large yellow meshes which form the ground mass of all sulphides of iron. Fig. 7 shows this appearance. A few grains of metallic iron only can be detected. It may, therefore, be affirmed that the yellow meshes are made up of the proto-sulphide of iron, FeS. As the temperature increases, the proportion of sulphur expelled also increases, and these products containing less sulphur are those which have been described as sub-sulphides. The micrographical examination, and even the simple examination with a magnifying glass show that they are mixtures of sulphides with a relatively high quantity of metallic iron.

These samples, however, present an interesting characteristic:

The iron which they contain is crystallized in dendrites having, according to the orientation, a very marked hexagonal or rectangular symmetry.

The Figs. 8 and 9 are good instances of this appearance. They, however, only show the centers of the crystallites whose branches have in reality a development two or three times greater than could be shown in the photographic field.



This hexagonal symmetry appears to indicate that iron, in its stable variety at a high temperature, belongs to the rhomboid

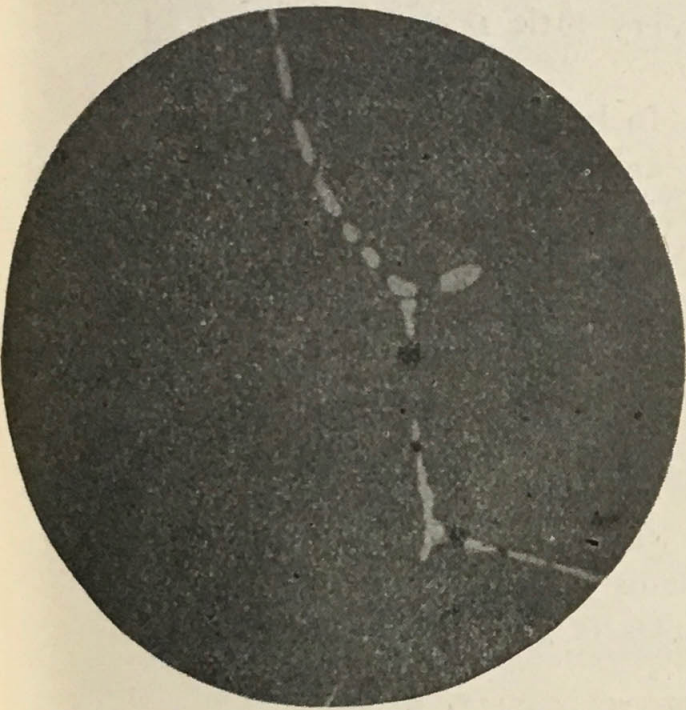


Fig. 7. Magnified 650 diameters.

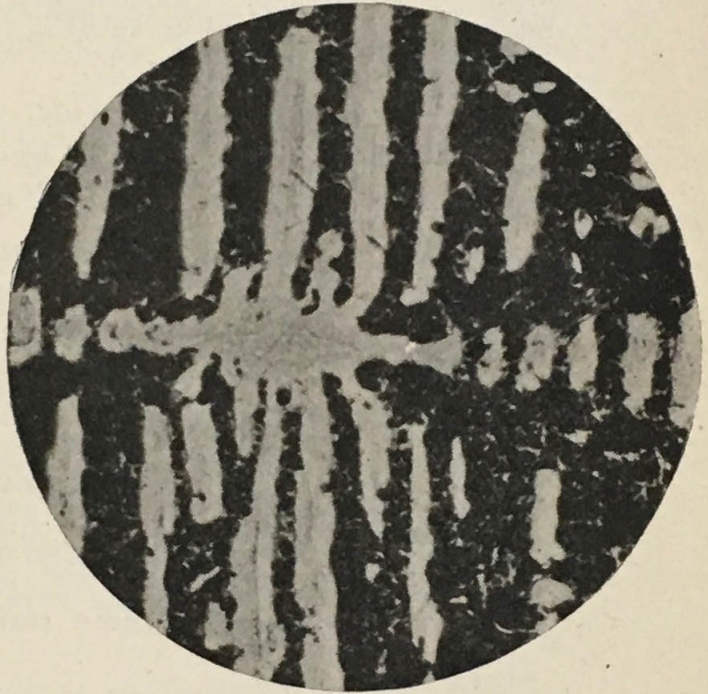


Fig. 8. Magnified 125 diameters.

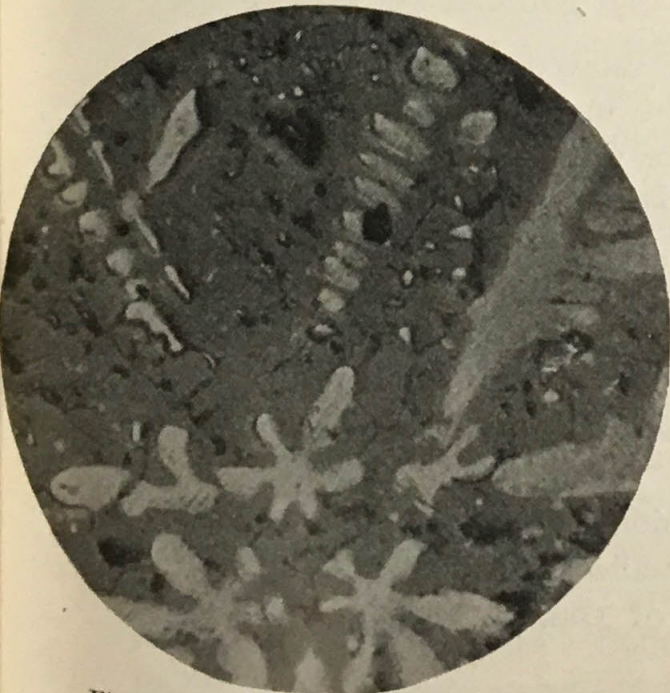


Fig. 9. Magnified 125 diameters.

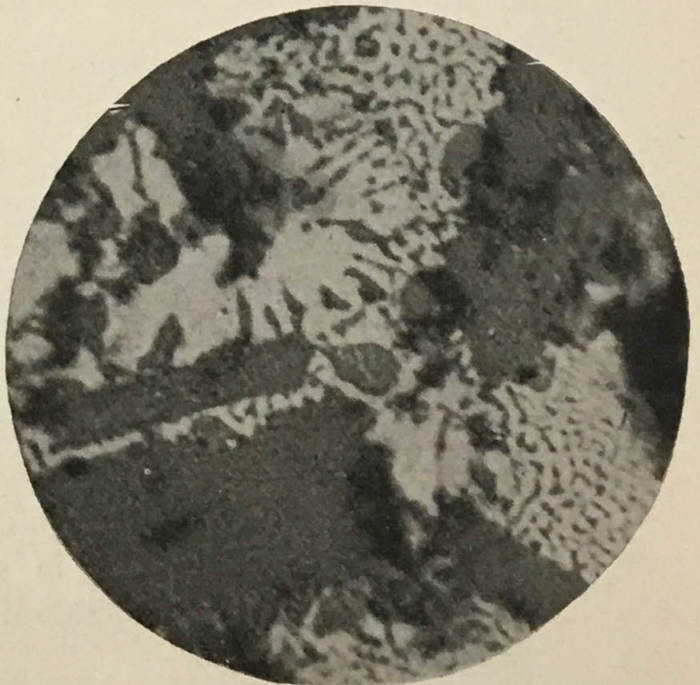


Fig. 10. Magnified 650 diameters.

system, and not to the cubic system. The existence of geometrical forms of crystals of iron indicates that in these mixtures,



rich in iron, it is this metal which begins first to solidify: iron is the element of first consolidation.

A diminution of pearlite is also observed in the sulphides heated to a high temperature. Very little pearlite is found in mixtures heated to 1500° C.

*Oxidation of the Sulphide.* — In heating the sulphide in air, sulphurous acid is expelled, and at the same time some oxide of iron is formed. It is the origin of the black crust which forms at the surface of pieces which have been heated and cast in air, as is the case with the sulphide sold by dealers in chemicals. It seemed likely that the gray constituent of the eutectic consists of this oxide. To verify this point, some melted sulphide was kept in contact with air for two hours, and the experiment was repeated at various temperatures.

The buttons obtained in this way were treated with bromine and left an abundant residue exclusively made up of oxide of iron. Composition of these samples is given below:

Sulphur	.	.	.	.	.	.	10.61
Iron	.	.	.	.	.	.	27.89
Insoluble Residue	.	.	.	.	.	.	61.24
							<hr/> 99.74

A micrographic examination showed that the pearlite was more highly developed than in the untreated sulphide.

Large elongated crystals were also detected (Fig. 10) and evidently correspond to the constituent of first consolidation. Its color was very similar to the gray component of the eutectic.

These experiments show that sulphide and oxide exist together and therefore afford a very simple explanation of the constitution of the eutectic.

A difficulty, however, remains which we have not succeeded in completely overcoming. Through the action of copper sulphate on the untreated sulphide some copper is deposited, although more slowly and in smaller quantity than upon iron (Fig. 11). Such precipitation of copper by a metallic oxide cannot be explained.

We have further verified that the proto-oxide of iron prepared by calcination of the oxalate does not precipitate any metallic copper, but some blue hydrated oxide. It is probable that the gray part is not homogeneous, but is itself, as should be

expected, a eutectic made up of a mixture of iron and of oxide, whose components, however, are too fine to be distinguished under the microscope. If this explanation is not accepted, it must be inferred that this gray substance is a solid solution of metallic iron and of oxide which contributes to the properties of both constituents.

We have, moreover, directly ascertained that this gray constituent is undoubtedly composed, for the most part, if not wholly, of oxide.

Some sulphide was melted in a current of dry hydrogen, which is without action on the sulphide, but which, on the contrary, reduces the oxide. In this way we caused the nearly complete disappearance of the eutectic. Fig. 12 shows the results obtained by heating in hydrogen some sulphide, whose initial structure is reproduced in Fig. 1. The dark areas appear to be made up of large grains of iron which were torn away in the polishing operation, leaving in their stead some cavities, which appear black in the photograph.

*Permeability of Iron by Sulphide of Iron.*—Mr. Campbell, and after him Mr. Arnold, have stated that at about  $1100^{\circ}$  sulphide of iron was capable of completely passing through soft steel or iron without leaving any trace of its passage in the metal. So surprising a statement should be repeatedly confirmed before being accepted. This consideration induced Professor Arnold to repeat the experiment of Mr. Campbell, the accuracy of which he doubted, and it is with the same end in view that we have again taken up this matter. We believe, however, that we have now conclusively shown the inaccuracy of the statement just alluded to.

Campbell and Arnold placed some sulphur, or better, as they say, some oxysulphide of iron, in a block of soft steel into which a hole had been drilled and then closed up by a plug of the same metal. This block was heated for one or two hours in a porcelain tube, and after cooling was cut in two lengthwise. It was found that the cavity was nearly always empty, although the plug was perfectly tight. Some sulphur was found, moreover, in the porcelain tube, near the lower part of the steel block, but in analysing the steel no increase in sulphur could be detected.

We repeated this experiment with exactly the same results. The cavity originally filled with sulphide was found completely



empty at the end of the operations, and its walls were as smooth as before introducing the sulphide. The experiment was repeated



Fig. 11. Magnified 650 diameters.

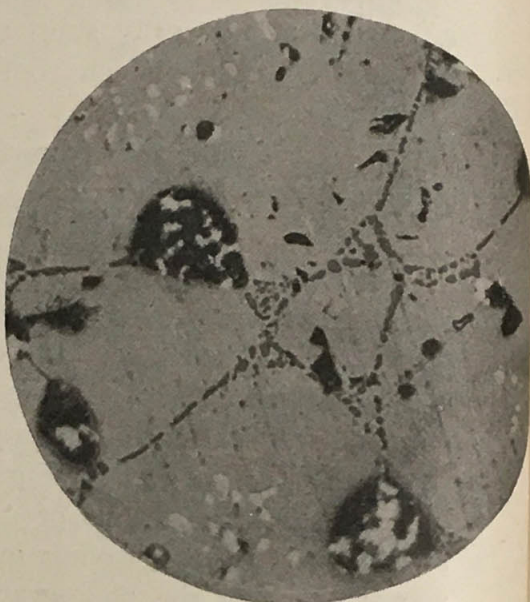


Fig. 12. Magnified 650 diameters

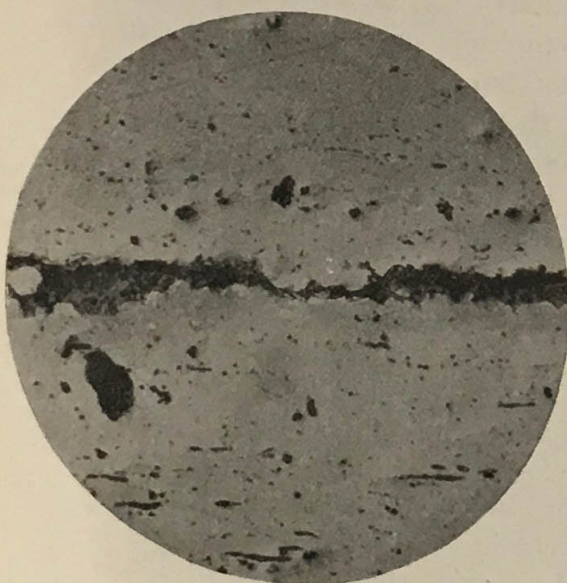


Fig. 13. Magnified 125 diameters.

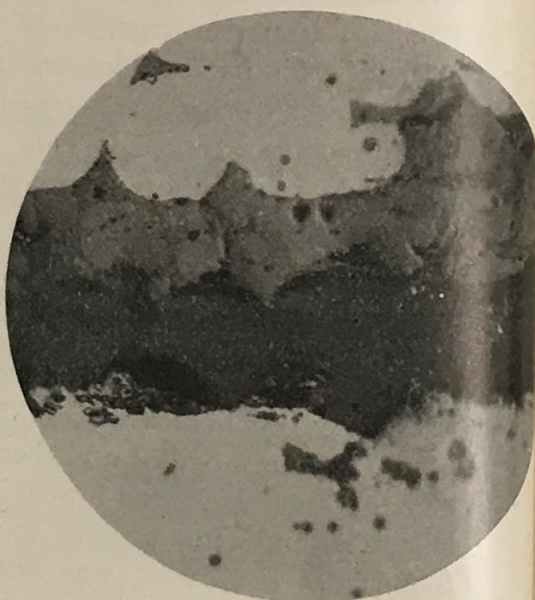


Fig. 14. Magnified 650 diameters.

in a bath of melted sodium chloride, and the results were the same. This bath prevents the oxidation of the sulphide and facilitates the collection at the bottom of the crucible of the sulphide



which had escaped, free from oxidation. These experiments apparently confirmed fully the conclusions of Campbell and Arnold.

To be certain, however, of the tightness of the joint we polished a longitudinal section of the block around the axis of the plug, and when examined under a microscope it was found that the joint was filled with sulphide along its whole length, although in many points these sulphide threads were less than 0.01 of a millimeter thick.

Fig. 13 shows a photograph of this joint. If the block be made of wrought iron the sulphide also finds a passage along the slag fibres which the metal contains. Fig. 14 is an instance of this. The large black area corresponds to the slag, and some sulphide, lighter in color, can be seen close to it. It is evident, therefore, that owing to its extreme fluidity the sulphide had simply escaped through the joints. The experiment was repeated in drilling a conical hole in the upper part of the block after insertion of its plug; the block was then heated in such a way that if the sulphide did escape from the joint it would find its way into the upper cavity and no longer run toward the lower part of the block as in the preceding experiment. The expected results were obtained: the expelled sulphur was found in the upper cavity where it could only have arrived by passing through the joint.

The escaping of sulphur through such tight joint may be accounted for if we consider that this substance is not only very fluid, but that it also dissolves a notable amount of iron, increasing thereby the opening through which it has begun to pass and facilitating the passage of the molten sulphide. The sulphurous acid, moreover, resulting from the reaction of the sulphide upon the oxide produces a pressure which has a tendency to expel all the substance contained in the enclosed cavity. This explains why the escape is easier with the oxysulphide of Mr. Campbell, which in reality is only a mixture of sulphur and of oxide, for in that case the production of the sulphurous acid is still greater.

*The Condition of Sulphur in Iron.* — Samples of iron containing only a few per cent of sulphur were prepared by one of the two following processes. In one instance we reduced by means of aluminum, as in the Goldschmidt method, some mixtures of iron and of sulphate of calcium in such proportion as to



yield samples containing from one to five per cent of sulphur. In reality, however, the preparations obtained do not correspond

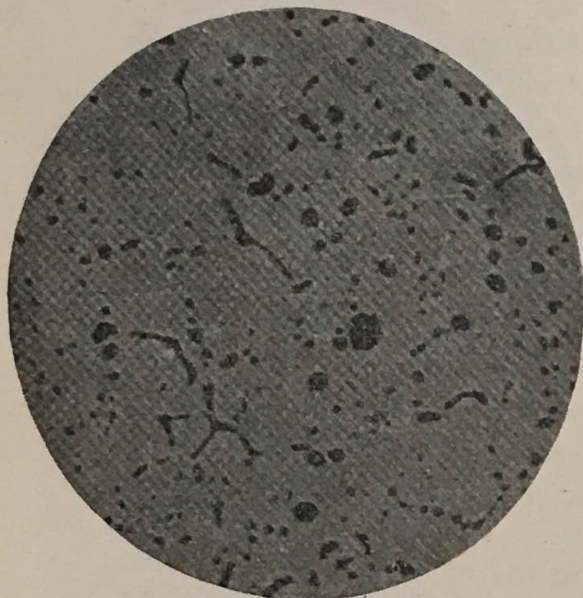


Fig. 15. Magnified 125 diameters.

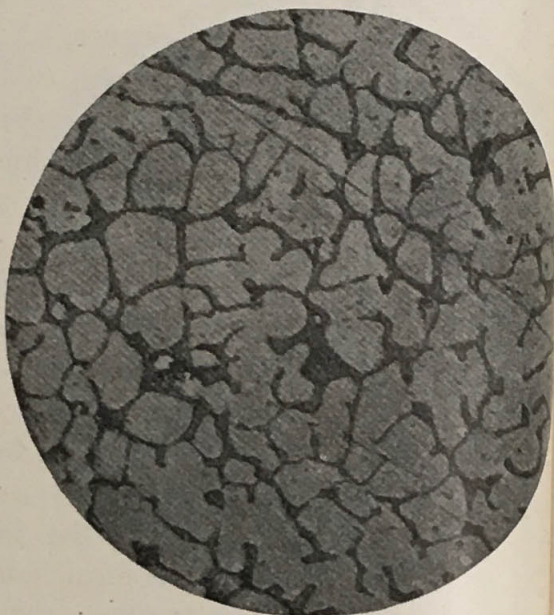


Fig. 16. Magnified 125 diameters.

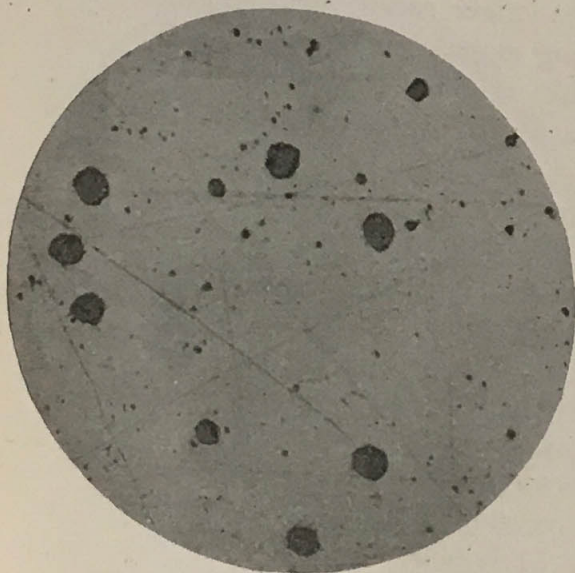


Fig. 17. Magnified 650 diameters.

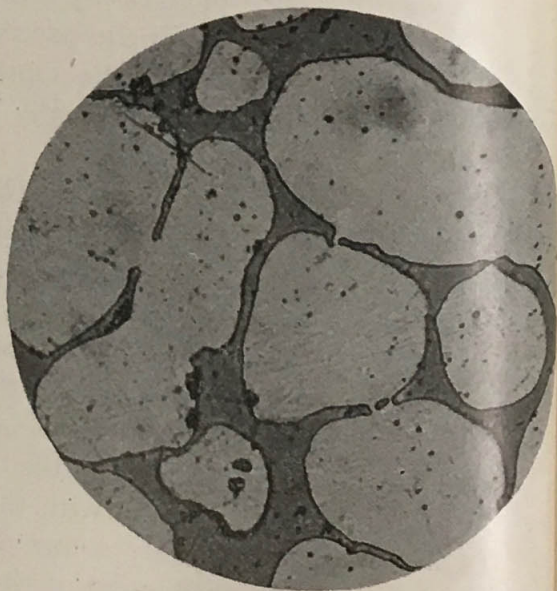


Fig. 18. Magnified 650 diameters.

exactly to the amount of material used, and they were in every case analysed. The second method consisted in melting a mixture of iron sulphide and of metallic iron by means of an acetylene blow-pipe. It was shown by one of us long ago that it is pos-



sible to obtain, by the combustion of acetylene with oxygen, temperatures reaching  $4000^{\circ}$  and so to regulate the character of the flame as to make it at will reducing, neutral or oxidizing without much variation of temperature. All the samples of iron obtained by these methods presented the same appearance.

All the metallic buttons obtained had a similar appearance. With less than one per cent of sulphur, the sulphide of iron is found, after polishing, mixed with a little of its eutectic, in the shape of round spots at first which become elongated as the percentage of sulphur increases. With a higher percentage of sulphur these areas are still more elongated and show a tendency to form a network. They constitute a membrane around the crystallized grains of iron. Figs. 15 and 16 show the appearance of two such structures, one containing 0.3, and the other 41 per cent of sulphur.

It can be readily conceived that these networks of sulphide, a substance having very little tenacity and melting at about  $950^{\circ}$ , will decrease considerably the resistance of iron in the cold, and completely destroy it when hot. We have found no certain indication of the existence of a solid homogeneous solution with small percentages of sulphur, as is the case with phosphorus.

We have, however, observed, that when the sample is etched by a suitable reagent, as, for instance, with the excellent reagent suggested by Mr. Igevsy, and consisting in a solution of 5 per cent of picric acid in absolute alcohol, the portions of the iron surrounding the sulphide are more quickly corroded, thus suggesting that they are less pure. When the sulphide, moreover, is present in isolated grains, such etching causes the appearance of two kinds of outlines. The first ones are found inside the polygonal figures, indicating the grains of sulphide; they are the normal outlines of the grains of iron. Much thicker lines, however, are also found, joining together these sulphide grains so as to completely trace the polygonal figures of which the sulphide areas occupy the summits. In Figs. 19 and 20 are shown the results of such an etching of the same samples, which, before etching, had the structure shown in Figs. 17 and 18.

It would seem, therefore, as if in the immediate surrounding of the sulphide and along the lines joining the sulphide areas, the metal possesses some special properties. For the present at least, we cannot speak more positively upon this point.



quickly. It is not certain, on this account, that the final equilibrium was attained.

The photographs of some of these buttons are shown here. Fig. 24 shows a sample obtained by the Goldschmidt process. Some yellow grains of sulphide of iron are clearly seen, and in the midst of them some crystals of sulphide of manganese may be detected. Fig. 25 shows the structure of a sample prepared by the acetylene blow-pipe; it contains some crystals of sulphide of manganese and some globules of sulphide of iron.

Finally, Fig. 26 shows, in a metal less rich in sulphur, a distribution of the sulphide very similar to the distribution of the oxide of copper in metallic copper. The formation of the sulphide is not, therefore, completely prevented, but its distribu-



Fig. 24. Magnified 650 diameters.

tion appears to have been modified. The sulphide seems to have a tendency to segregate in the midst of the metal in the shape of isolated grains instead of forming a continuous network between the grains. It would be interesting to repeat these experiments with ingots of greater size, and cooled as they are cooled in practice, in order to ascertain whether the distribution of the sulphur between the manganese and the iron is not modified.

*Sulphur and Nickel.* — It is well known that sulphur is especially dangerous in the case of nickel steel. It was, therefore, interesting to study the relations between sulphur and nickel. The sulphide  $\text{NiS}$  is known and appears quite stable — much more so than sulphide of iron. It has a very metallic lustre. Another sulphide has been described, the sub-sulphide  $\text{Ni}_2\text{S}$ . We have endeavored to prepare this last compound in melting

the required quantities of nickel and of proto-sulphide. The fracture of the resulting button had an homogeneous metallic



Fig. 25. Magnified 650 diameters.

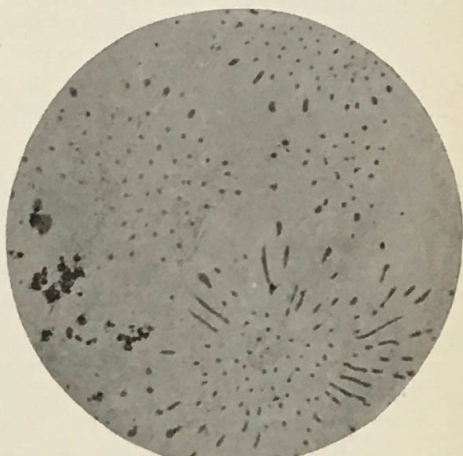


Fig. 26. Magnified 650 diameters.

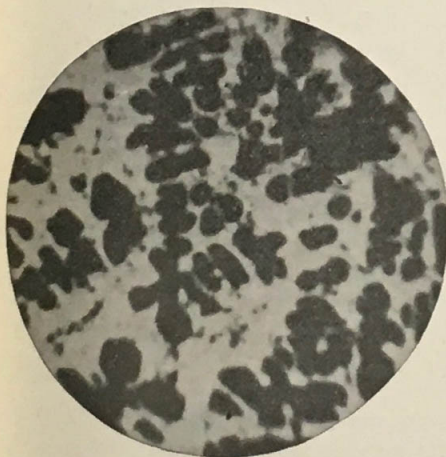


Fig. 27. Magnified 125 diameters.

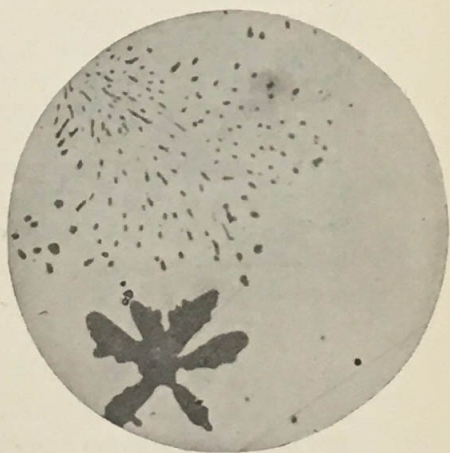


Fig. 28. Magnified 650 diameters.

appearance, as was also the case with the polished section. It is owing to this appearance that this product was considered as a definite compound. Such an argument, however, is without



value, if we consider the metallic lustre of the proto-sulphide which makes it absolutely similar to the metal itself. In examining this polished surface, however, under the microscope, the presence of two distinct elements is suspected, and they are revealed by etching the section. The sulphide remains bright, while the metal is quickly affected. This so-called sub-sulphide is merely a mixture of nickel and of proto-sulphide.

Fig. 27 was obtained in etching a sample assisted by the electric current generated by a battery, during a very long time, so that the metal was dissolved to a considerable depth. The cavities, resulting from the solution of the nickel, were then filled with a black varnish. The sample was again polished and photographed. It is in this way that the great contrast shown in the photograph was obtained.

Some ferro-nickel, containing 25 per cent nickel and 1 per cent manganese, melted together with a little sulphide of iron by means of the acetylene blow-pipe gave a structure shown in Fig. 28, and in which the sulphide of manganese may readily be detected under its two different aspects.

We have not in this research dealt with carburetted metals. We may, however, mention the fact that, very frequently, very regular crystals are found in gray cast iron, which, owing to their characteristics, resemble crystals of sulphide of manganese. These crystals have a less dendritic appearance than those which we have observed, but the slowness of the cooling of these industrial products suffices to explain this difference of appearance, for it is known that the slower the formation of the crystals, the nearer they approach their normal forms, convex polyhedra.

SHORT REPORTS FROM THE METALLURGICAL AND  
METALLOGRAPHICAL LABORATORY OF THE ROYAL  
MECHANICAL AND TECHNICAL TESTING INSTITUTE  
OF CHARLOTTENBURG \*

By E. HEYN

THE following short reports are made as a contribution towards answering the question as to in what measure metallographical results and processes can find application for the purpose of testing material. A review of the whole sphere of applicability is not intended, and indeed could not be given in the form of a short lecture. Only some few examples which lie ready at hand will be selected for consideration. The important rôle which metallographical experiments fill when it is sought to determine the nature of the heat-treatment which the specimen of material has undergone, will here be passed over, as a review of this matter has been supplied at the Stockholm Congress by the eminent specialist Mr. Osmond.

Another very extensive branch of metallography, the determination of the course of permanent deformation produced in metals at the ordinary temperature, will only be touched upon in two special cases, as the same investigator, Mr. Osmond, has announced a lecture on the subject to be given before this assembly.

The labors of the Testing Institute in this department† are published in the Journal of the Association of German Engineers (*Zeitschrift des Vereins deutscher Ingenieure*, 1900, Nos. 14 and 16). I should first like to direct your attention to the action of hydrogen on iron and copper, and to the action of oxygen on copper.

I. IRON AND HYDROGEN.

In the chemical analysis of iron and steel two elements, viz. hydrogen and oxygen, are almost entirely disregarded, not because it is supposed in advance that these elements have no injurious influence, but rather because the analytical determina-

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\* *International Association for Testing Materials.* Budapest Congress, 1901.

† E. Heyn. The changes in the microstructure of iron and copper produced by cold-work and subsequent heating.



tion of these substances either is connected with considerable difficulties, as in the case of hydrogen, or no reliable methods of experimenting have yet been worked out, as is the case with oxygen. The injurious influence of oxygen on iron is generally known and feared. But, unfortunately, practically nothing is known as to the form or forms under which it exists in iron.

Concerning the effect of hydrogen on the properties of etched iron we have been enlightened by the researches of Hughes and Ledebur on brittleness caused by corrosion. But that hydrogen is capable also of effecting iron at red heat and that the phenomena thereby produced can be extremely unpropitious in testing materials may not be generally known.

The researches carried out in the Testing Institute by the author in this connection were occasioned by the irregularities which were perceived in the course of practical bending-tests with quenched rods. Sometimes it occurred that soft mild steels supported the bending tests with quenched rods to perfection, that is to say, after being heated to red heat and quenched in water, they could be completely bent, without disclosing cracks, if quenching was effected after heating in a smith's fire; but on the other hand, the same material failed to support the test and tore even when bent to a small degree, if before the quenching the heating was carried on in a muffle-furnace heated by coal-gas. It was then ascertained that defective draft in the furnace and lengthened period of heating exercise an unfavorable influence on these tests. There was no increase in the carbon contents of the steel.

This is a surprising phenomenon, and I am convinced that in testing practice the experimenter was repeatedly struck by the puzzling fact, that one and the same material withstood the bending test or not, according to the manner in which the previous heating was carried out, even if particular pains had been taken as to the constancy of the temperature, from which quenching was done. It appeared from observations made in the Testing Institute, that the origin of this phenomenon is to be sought for in the *hydrogen* contained in the coal-gas, which naturally could only come in contact with the specimen experimented upon in case an imperfectly closed furnace-muffle were employed, and in case of an incomplete consumption of the coal-gas by reason of defective supply of air. The results of the experiments in

connection with the action of hydrogen on iron have been published in detail by the author of this article in the journal *Stahl und Eisen*, 1900, No. 16.\* In what follows only the principal points will be mentioned.

a. If mild steel be heated in contact with hydrogen to a temperature between  $730^{\circ}$  and  $1000^{\circ}$  C., and immediately afterwards plunged into water, the metal becomes considerably more brittle, than if the heating which precedes the action of immersion had been carried on in the air. Slow cooling in hydrogen as well as immersion in water after heating the metal in hydrogen to a degree lower than  $730^{\circ}$  C. produces no effect, which can be demonstrated by a bending test.

The degree of brittleness produced was determined by means of bending-tests on specimens of sections  $8 \times 8$  and  $9\frac{1}{2} \times 9\frac{1}{2}$  mm. respectively. The edges of the rods tested were intentionally not rounded off in order that the influence of the brittleness could more effectively manifest itself. The bending was produced in all cases on a dome of 8 mms. radius. Specimens of soft basic Martin steel with .05 C, less than .01 Si, .37 Mn, 0.62 P, 0.46 S, .03 Cu, which, after being heated in the air to a tem-

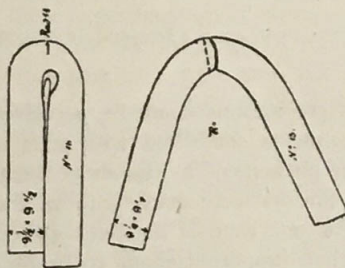


Fig. 1. Soft Martin-Steel.

- (a) Heated to  $820^{\circ}$  C in air, quenched in water of  $11^{\circ}$  C. Free from cracks when bent together flat.
- (b) Heated to  $820^{\circ}$  C in hydrogen, quenched in water of  $11^{\circ}$  C. Broken suddenly into 2 parts when bent to an angle of  $138^{\circ}$ .

perature between  $760^{\circ}$  C. and  $1000^{\circ}$  C. and then plunged into water could be bent together flat without showing cracks, broke suddenly if bent to a small angle in case the heating before im-

\* E. Heyn, Eisen und Wasserstoff.



mersion had been carried out in hydrogen under otherwise similar conditions. The specimens exhibited are evidence of this. Figs. 1 and 2 are sketches of the bent specimens in half their natural size. They are selected in order to give some idea of the influence of hydrogen.

The cracks in the specimens made brittle by means of hydrogen appeared always on the side of the tension. The tearing took place instantaneously with sharp cracking even after

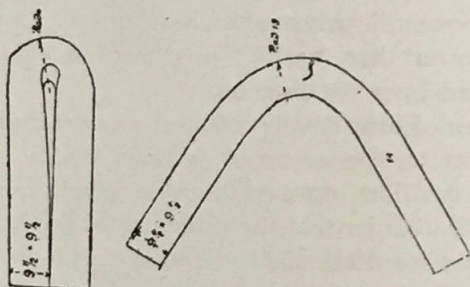


Fig. 2. *Soft basic Martin-steel.*

- (a) Heated for an hour in air at a temperature  $760-780^{\circ}\text{C}$ . Quenched in water of  $13^{\circ}\text{C}$ .
- (b) Heated for an hour in hydrogen at a temperature  $760-780^{\circ}\text{C}$ . Quenched in water at  $13^{\circ}\text{C}$ .

several occasional crepitations could be perceived in the broken specimens. Experiments conducted with steel used for girders produced the same phenomena as the above-mentioned soft steel.

Further experiments were made with wire of two different sorts of steel. The one wire of diameter 3.7 mm. and containing very little carbon supported about 16 bendings, each of  $90^{\circ}$ , before breaking, and this after being heated in air for half an hour at a temperature of  $820^{\circ}\text{C}$ . and subsequently quenched in water. The same wire broke after 9 bendings when the heating before immersion had been carried out in hydrogen, but otherwise under similar conditions. In the case of a somewhat longer sustained heating in hydrogen at the same temperature the quenched wire supported only  $2\frac{1}{2}$  bendings.

A harder wire of 3.9 mm. diameter and 0.37 per cent carbon, after being heated in air for half an hour at a temperature of  $800^{\circ}\text{C}$ . and then quenched in water, could be bent to an

angle of  $64^{\circ}$  without breaking, whereas, after being heated in hydrogen and quenched in water, the same wire could support bending only to  $34^{\circ}$ .

*b. The action of the hydrogen penetrates gradually from the surface to the interior of the specimens.* This is evidenced by the following selected experiments carried out with the before-mentioned mild Martin-steel. The heating took place in hydrogen at a temperature of  $800^{\circ}\text{C}$ . From one specimen, a layer of 1.2 mm. in thickness was planed off on the surface to which the tensile forces were to be applied afterwards. This was done before the heating and quenching took place. The specimen was bent to an angle of  $150^{\circ}$  and then cracked suddenly.

The second specimen was treated in exactly the same manner with the difference only, that the layer of 1.2 mm. thickness was planed off after the process of heating in hydrogen and quenching in water. It could be bent into a knot without breaking. By removing the upper surface layer made brittle by the action of hydrogen, the capacity of sustaining bendings without breaking was considerably increased. It appears from this, that bending-tests with notched specimens are not suitable for showing the action of hydrogen, if the notch is made after heating and quenching. By these experiments it is explained also, why bodies with small section show the influence of hydrogen after being heated for a shorter length of time than bodies with a greater cross-section, because in these latter the hydrogen has a greater distance to penetrate into the interior.

*c. The brittleness of steel caused by heating in hydrogen and quenching in water can be weakened or removed entirely by subsequent heating in air or nitrogen.* If the action has not penetrated very deep, it is sufficient to boil the specimen in water to produce a perceptible decrease of brittleness. It must here be mentioned that for the purpose of comparison always specimens were selected which had been heated in air under exactly the same conditions, then quenched in water and afterwards subjected to the same treatment as the specimens which had been heated in hydrogen, in order that the effect of tempering could be taken into calculation.

Heating to a temperature of  $200^{\circ}$  to  $250^{\circ}\text{C}$ . is sufficient in the case of materials poor in carbon to remove the effect of the hydrogen. The specimens receive the same properties as



those specimens treated in like manner which had been heated in air before being quenched in water. In the case of materials rich in carbon a decrease in brittleness is brought about by this treatment, but still not the same degree of toughness is obtained as is shown by a specimen of the same steel heated in air, quenched in water, and tempered to the same temperature. The degree of toughness is nearly in the proportion of 2:5. It appears from this as if materials rich in carbon retain the influence of hydrogen more obstinately than those which are poor in carbon.

At all events, by heating in nitrogen or air to red heat, the brittleness due to hydrogen is removed.

*d. Lengthened exposure to the air of specimens heated in hydrogen and then quenched in water diminishes or totally removes the brittleness.* In bending-tests with specimens of greater cross section in which presumably the action had not penetrated deep inwards, a few days' exposure to the air at ordinary temperature was sufficient in order to obtain considerably more favorable results. On the other hand in the case of wires of small cross section a considerable period of exposure to the air was needed in order to diminish the brittleness occasioned by the action of hydrogen. In Fig. 3 the effect of exposure to the air is represented in a diagram for a mild-steel wire of 3.7 mm. diameter, very poor in carbon.

The line *a* gives the constant number of bendings  $14\frac{1}{2}$  for the wire heated in air and then quenched in water.

The line *b* gives the number of bendings gradually increasing with the period of exposure to the air, for wires which were heated the same time in hydrogen and then quenched in water. These specimens were bent after intervals of time represented by the abscissæ in Fig. 3. As is shown, even after 250 days' exposure the brittleness produced by the action of hydrogen was not completely destroyed, although very considerably diminished.

It ought to be observed that the behavior of steel heated in hydrogen and then quenched in water is as in general so also in particular with regard to a lengthened exposure to the air completely analogous to the behavior of iron which, according to the researches of Ledebur in connection with brittleness, has obtained its hydrogen through the action of diluted acids at ordinary temperature.

*e. The microstructure of the specimens heated in hydrogen*

and then quenched in water did not show the slightest difference from the structure of those heated in air and then quenched in water. Also no difference in specific weight could be perceived by means of the method employed.

Experiments were made on the specimens which had been heated in hydrogen and then quenched for the express purpose of examining if hydrogen was still contained in them. As the influence of hydrogen is destroyed at red heat, heating to this degree should have been sufficient in order to expel the element producing brittleness. Nitrogen was passed over the heated specimen; the hydrogen expelled passed over red-hot copper oxide to be subsequently weighed as water in a tube containing

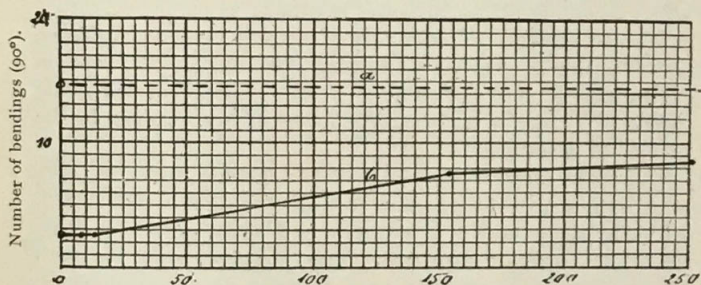


Fig. 3. Time of exposure of quenched specimens to the air.

- (a) Specimen heated in air for half an hour at  $820^{\circ}$ , then quenched in water.  
 (b) Specimens heated in hydrogen for half an hour at  $820^{\circ}$ , then quenched in water.

phosphoric acid. In employing a specimen of the same dimensions as those in Figs. 1 and 2 and possessing a weight of 83 gr. there was an increase of only .0015 gr. in the weight of the tube containing phosphoric acid. An experiment without steel specimen gave an increase of weight of .0002 gr.

Repeated experiments with specimens embrittled by hydrogen gave always values differing very slightly from .0015 gr. This weight would correspond to a percentage of .0002 by weight of hydrogen contained. The weight .0015 gr. is, in view of the somewhat complicated conditions of the experiment, not sufficiently different from the possible deviations due to analytical errors as to be accepted as a direct proof that hydrogen was



really contained in the quenched specimens. It only proves that hydrogen was probably present in the specimens. But at all events analytical determination proves that greater quantities of hydrogen than .0002 per cent in weight are not present in the brittle specimens.

The objection is still to be answered, that the steel which has become brittle after heating in hydrogen and then quenching in water gives off on being heated again absolutely no hydrogen, but that the hydrogen which has already been absorbed remains, assuming a dangerous form during the process of quenching and changing back again into a harmless form during the reheating. If this be the case, hydrogen would behave like carbon which according to circumstances is present as hardening carbon or carbide carbon. It could further be said that this hydrogen which cannot be expelled by heating does not directly occasion the brittleness, but only if it is in the presence of hardening carbon or of one of the allotropic forms of iron retained by the process of quenching.

In both cases the iron which has become brittle by once being heated in hydrogen and then quenched in water should, on being again heated in nitrogen and again quenched, show the original state of brittleness. This, however, is not the case; the iron after this treatment receives again its normal qualities.

It follows from this that the two possibilities mentioned are excluded and that, as a matter of fact, the hydrogen which occasioned the brittleness in the specimens is expelled by the subsequent heating in nitrogen. Consequently, the pernicious factor must reside in the above-mentioned small quantity of .0002 per cent by weight of hydrogen. This is not difficult of belief when we take into consideration that already .028 per cent by weight of hydrogen can render iron produced by electrolysis as hard as glass, and that the brittleness observed by Ledebur in corroded steel is produced by .002 per cent. It would, therefore, not be very surprising if the tenth part of this amount exercised the powerful effect as above described on steel which has been quenched in water.

In explanation of this phenomenon I should suggest as follows: At temperatures varying from  $730^{\circ}$  to  $1000^{\circ}$  C. steel absorbs hydrogen in small quantities. Below  $730^{\circ}$  C. the capacity of absorbing diminishes and, in consequence of this, if the

steel is slowly cooled in hydrogen, nothing of this gas is retained; with decreasing temperature the hydrogen leaves the steel.

If, on the other hand, the steel, after having absorbed hydrogen at the above-mentioned temperature, is suddenly quenched in water, the hydrogen cannot escape fast enough, but is retained mechanically. It is, however, in a state of unstable equilibrium which is disturbed by heating even to a low temperature, and this produces the escape of the hydrogen. The absorption of hydrogen at red heat takes place from the surface inward. In support of this theory of absorption we have also the fact, that a specimen of steel which had been first heated half an hour in hydrogen and directly afterwards under conditions remaining the same heated in nitrogen and then quenched in water, did not become brittle. The nitrogen expels the hydrogen again. Consequently it follows, that hydrogen enters and escapes at the same temperature. This process takes place in the direction from outwards into the steel if the atmosphere surrounding the specimens contains so much hydrogen that its partial pressure causes the hydrogen molecules to enter into the iron. The process takes place in the reverse direction if, on the disappearance of hydrogen and its replacement by nitrogen, the hydrogen partial pressure in the atmosphere surrounding the specimen has become equal to nill, in which case gas escapes from the iron.

From the researches of St. Clair-Deville and Troost as well as from those of Graham, 1866, it is known that hydrogen can diffuse through red-hot iron. Perhaps the molecular changes produced in iron at a temperature of  $700^{\circ}$  to  $1000^{\circ}$  C. facilitate the diffusion. By quenching in water a specimen which has been saturated with diffused hydrogen, the hydrogen can be retained in unstable condition. Perhaps this accounts for the extremely small quantity of injurious hydrogen.

A question of importance which, however, still requires nearer explanation is, how the hydrogen which is retained in mild steel on *solidification* conducts itself, and especially what rôle this hydrogen plays when the specimen is hardened. Axel Wahlberg has made the observation (see *Jernkontorets Annaler*, 1900, "Om Kisels inverkan på ståls hållfasthetsegenskaper") that mild steel blown and cast at a too high temperature, if it contains more than the usual quantity of silicon, on hardening



becomes more brittle than mild steel blown and cast at a normal temperature the silicon contents of which were made as high as in the former case by adding silicon at the end of the blowing process. Wahlberg is also of the opinion that this is to be ascribed to the presence of alloyed gases.

## 2. COPPER AND HYDROGEN.

Hydrogen has still more deleterious influence on heated copper than on iron. If copper be heated to a temperature above  $600^{\circ}\text{C}$ . in an atmosphere of hydrogen this gas exerts its influence from the surface inwards. The surface-layer becomes extraordinarily brittle and shows shell-like fractures of a yellowish-red color. With the duration of heating and the degree of temperature the thickness of the transformed layer increases until finally the copper core entirely disappears. The specimens exhibited show this in different stages. The specific gravity diminishes considerably under this treatment, viz., from 8.9 to 8.4. The surface of the copper heated in hydrogen at temperature of  $800^{\circ}\text{C}$ . showed already before being hammered in a cold condition fine longitudinal hair-like fissures (*Comp. Zeitschrift des Vereins deutscher Ingenieure*, Journal of the Association of German Engineers, 1900, Nos. 14 and 16, E. Heyn, "The changes in the microstructure of iron, copper," etc.). The microscopic texture is changed. This phenomenon might be explained by the circumstance that the copper oxidule or other oxides contained in small quantities in all copper found in commerce are reduced by the hydrogen on heating to red heat. In consequence of the difference between the volume of the original oxide and that of the metal formed by the reduction of the oxidule, small cavities filled with gas can arise and affect the brittleness of the metal. A rough calculation shows, however, that the considerable difference in the specific weight before and after the treatment in hydrogen could be explained on the basis of this assumption only if very great quantities of oxides were in the copper. Even then the cracks on the surface of the heated copper would still remain unexplained. This question will be the subject of a more exhaustive investigation in the Testing Laboratory of Charlottenburg.

### 3. COPPER AND OXYGEN.

The action of copper and oxygen on each other has been determined by the researches published by the author in the *Reports of the Royal Technical Testing Institute*, Charlottenburg, 1900, p. 315,\* so far as the action is limited only to copper and oxygen. The conditions become more complicated when occasion is given for the formation of arseniates and antimonates. Experiments in this connection are to be continued.

In view of the great chemical affinity between copper and oxygen, these two elements can only exist together in the chemical compound  $\text{Cu}_2\text{O}$ , copper-oxidule, when there is an excess of copper.

Copper-oxidule forms with copper in the liquid state a homogeneous solution in the manner of salt solutions; the reciprocal solubility is, to a certain extent, perfect. As a consequence of solidification the solubility of copper-oxidule in copper becomes practically null; the process of solidification brings about a separation of the copper from the copper-oxidule. In the texture of the solidified alloy copper and copper-oxidule should be perceptible as separated elements of the texture, which in reality is the case. The course of the solidification of the alloy of copper-oxidule in copper is represented in the freezing-point curve, Fig. 4. In this figure the abscissæ represent the quantity of copper-oxidule in the alloys, and the ordinates the temperatures, measured with the help of the pyrometer at which the solidification began and ended. Between these two temperatures lies the course of solidification for the alloy under consideration.

A distinct point of solidification or of melting is only observed in the case of pure copper or in the case of an alloy with 3.4 to 3.5 per cent copper-oxidule, that is, the so-called eutectic alloy. In all other alloys solidification takes place between two limits of temperature, the lower of which corresponds to the point of solidification of the eutectic alloy. The upper limiting point, that is, the beginning of solidification, rises on both sides, starting from the eutectic alloy's freezing point and tends, on the side of mixtures weak in oxides, towards the point of solidification of pure copper and, on the other side, to the point of solidification of pure copper-oxidule. The microstructure of

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\* Heyn, Copper and Oxygen.



copper which contains small quantities of oxidule consists only of microscopically small copper-crystals, as shown by the sketches in Figs. 15 and 16. The texture of the eutectic alloy with 3.4 to 3.5 per cent copper-oxidule consists, as is in general the case with eutectic alloys, of an extremely intimate mixture of small copper and copper-oxidule crystals, as can be seen from micro-photograph 18 magnified 123 times. The microstructure of alloys with 0 to 3.4 per cent oxidule consists of round copper crystallites (*k*) surrounded by the intimate mixture of copper and copper oxidule corresponding to the eutectic mixture, (*e*), micro-photograph 17. If the oxidule contents exceed 3.5 per cent, the copper crystallites (*k*) disappear, and in place of them appear finger-shaped crystals of copper-oxide which lie embedded in the eutectic mixture (*e*), micro-photograph 19.

During the solidifying process of alloys with less than 3.4 per cent, oxidule copper crystals first separate out from the liquid mass. The quantity of crystals increases, the quantity of mother-liquor diminishes, and with this is connected an increase in the proportion of copper-oxidule in the latter. This augmentation continues while the number of copper crystals continually increases and the temperature falls, until the mother-liquor has reached 3.4 per cent copper-oxidule, that is to say the proportion contained in the eutectic alloy, and then solidifies into an intimate mixture of small copper-oxidule crystals at the freezing point of the eutectic alloy.

In cases where the oxidule contents exceed 3.5 per cent the process is similar, but the crystals separating out from the mother-liquor are copper-oxidule; the mother-liquor becomes thereby poorer in oxidule, and finally, at the point of solidification of the eutectic alloy arrives again at the composition of the eutectic mixture 3.4 to 3.5 per cent. All alloys, therefore, when solidifying, conduct themselves in such a manner, that the part which remains liquid always approaches the composition of the eutectic alloy with 3.4 to 3.5 per cent oxidule. If the alloy contains an excess of oxidule, this must separate out; if there is less copper-oxidule, copper must separate out in order that the above condition may be arrived at.

By the separation of such crystals from the fluid mass, the alloy becomes thick in the course of the solidification process. This explains why copper containing oxidule is not so thin in

the fluid state as copper which is free from or weak in oxidule.

From Fig. 4 can be calculated what must happen with copper free from oxidule when it is heated in the air gradually to a temperature  $18^{\circ}\text{C}$ . below that of the melting point of pure copper. If heating is continued to the temperature corresponding to the ordinate  $OD$  in Fig. 4, the copper will take up oxygen

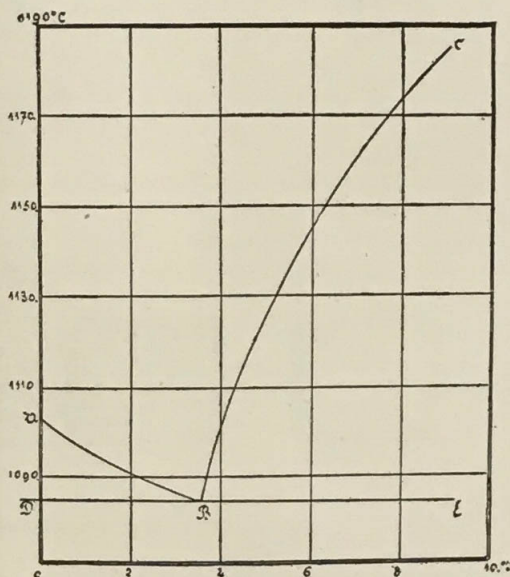


Fig. 4. Contents of copper-oxidule (%)

*Obs.* The temperatures are only relatively exact.

*AB* Beginning of separation of copper.

*BC* Beginning of separation of copper-oxidule.

*DE* End of solidification process. Eutectic line.

from the air, and with this form a fluid eutectic alloy lying distributed in the great mass of the metal, which remains in a solid state. During this there is no increase of temperature.

If the heating be continued long enough, the eutectic fluid part increases continually, and the remainder of the solid copper swims in it, forming a kind of paste. This continues further until the whole mass is transformed into the eutectic mixture



and is in a fluid state of aggregation. From this moment the temperature can again begin to rise. If the above proceeding be interrupted before the quantity of fluid eutectic alloy has reached its maximum, the copper retains its external form without any perceptible change. It is, however, no longer pure copper, but has become an alloy of copper-oxidule in copper which, after solidifying, does not show the same properties as before. At a temperature of  $18^{\circ}$  C. below its melting point, copper is able, therefore, to absorb oxygen in its entire mass, it becomes "burnt" copper. If, on the other hand, the temperature is lower, oxygen is still absorbed by the copper, but only on the surface in the form of a layer of oxidule; the interior mass remains unchanged.

The final results can be seen in Figs. 5, and 6, in which the shaded parts represent copper-oxidule.

Fig. 20 gives the microphotograph of a burnt copper treated as in Fig. 6, magnified 365 times. The specimen was not yet

Copper heated  
in the air below  
the eutectic tem-  
perature

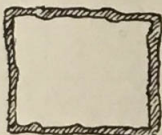


Fig. 5.

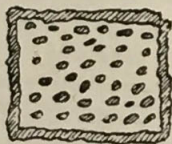


Fig. 6.

Copper heated  
in the air at the  
eutectic temper-  
ature ( $18^{\circ}$  C. be-  
low melting point  
of pure copper).

melted, had preserved its original shape, and its composition approaches that of the eutectic alloy.

Fig. 21 gives a photograph, magnified 123 times, of a polished surface from a small copper tube which had been used for overheating steam in a laboratory apparatus and which had been heated externally by a Bunsen burner. Here a similar phenomenon appeared as that which was above described. The copper without melting took up oxygen which, in the form of fine veins of oxidule, lay nestling among the grains of copper. The material had in consequence after a time become so brittle that it could be crushed with the fingers.

The direct influence of uniformly distributed oxidule on the resisting properties of the copper is not particularly evident, but tends to diminishing the toughness of the metal. According to Hampe (*Zeitschrift für Berg-, Hütten- und Salinenwesen im*

*Preussischen Staate*, 1874, 22, p. 94) the diminution in toughness becomes apparent when there is 2.25 per cent of oxidule. But here also analysis seldom gives correct information, as it leaves us in the dark as to the distribution of the oxidule in the specimen.

In the copper of commerce are found accumulations of oxidule in the different parts of the copper where the oxidule contents rise almost to 3.5 per cent, but the average contents of the whole specimen do not exceed the above-mentioned proportion 2.25 per cent. Such accumulations which can be perceived by means of the microscope, can exert considerable influence on the properties of the metal.

Thus, for example, a hard-drawn or hard-rolled copper rail, which was occasionally examined, showed numerous linear layers of copper-oxidule, lying lengthwise. The total oxidule contents amounted, according to estimation made by means of the microscope, to less than 1 per cent; in some places, however, in the line-like accumulations, it rose higher, almost to the eutectic contents. Microphotograph 22 shows, on the right, such a line of copper-oxidule, and on the left material poor in oxidule, magnified 123 times. The bar could be bent completely after being annealed, but in the original state in which it was cold-worked and then made use of, its bending capacity, in comparison with material weak in oxidule, which had undergone the same treatment, was considerably diminished.

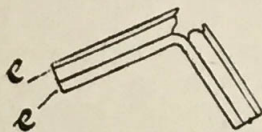


Fig. 7.

As Fig. 7 shows, the bar broke on being bent along the line-like oxidule layers.

It can be seen from this, that the influence of the oxidule on the properties of the annealed copper, even under unfavorable conditions of accumulation, as here, does not make itself much felt, but that in a cold-worked condition, when the capacity of the copper crystals for undergoing a change of form is already smaller, the unfavorable influence occasioned by the



presence of layers of the foreign body embedded in the copper becomes distinctly perceptible.

#### 4. TWO INSTANCES OF PERMANENT DEFORMATIONS AT ORDINARY TEMPERATURES.

The following example is calculated to show how extraordinarily sensitive are metallographical methods relating to the discovery of permanent deformations at an ordinary temperature (specimens cold-worked). From a 10 mm. thick, flat piece of steel consisting of the softest basic Martin material, two specimens (I. and II.) for bending tests were cut by halving the flat iron longitudinally as shown in Fig. 8.

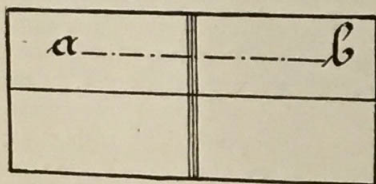


Fig. 8.

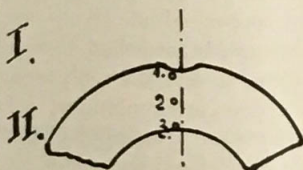


Fig. 9.

Both specimens were notched on one side. Specimen I was bent at the ordinary temperature over a dome of 10 mm. into the curvature shown in its natural size in Fig. 9. After the bending, the specimen was cut in the direction of the length *ab* in Fig. 8, the section thus obtained was polished and etched. The spots 1, 2, 3 marked in Fig. 9 were magnified  $\frac{38.5}{1}$  times and sketched as shown in Figs. 10, 11 and 12. From these figures the size and form of the grains of ferrite can be seen. The point 1 corresponds to the stretched side close to the notch, the point 3 represents the compressed side, and 2 is at the middle point between 1 and 3. From Figs. 10, 11 and 12 it can immediately be seen that the grains of iron on the side of traction are stretched in the direction of the specimen's length. On the compressed side the grains of iron are also stretched, but in a direction perpendicular to the longitudinal axis of the specimen. In the middle at point 2 no stretching of the grains is perceptible. It is to be remarked that in Figs. 10, 11 and 12 the sketches are arranged in a position corresponding to Fig. 9.

In cases of smaller deformation than in the above example, a simple inspection of sketches is not sufficient to ascertain beyond doubt the change of form in the grains. In this case, measuring will certainly lead to the attainment of this object. For the purpose of measuring, a system of lines not too far apart is drawn through the sketch as indicated in Fig. 10. It is, however, advantageous not to choose a circular limit, as in Fig. 10, which cuts the grains indiscriminately, but to arrange the limit so that only complete grains lie in the sketch. If it is desired to determine the average measurement of the grains in the direction  $Q Q$ , the whole length of a line, for instance  $xy$  (80 mm.), is measured. As the line cuts 10 grains on the average, there are  $\frac{80}{10} = 8$  mm. for each grain.

The same procedure is repeated for all the parallels to  $Q Q$ , and a total average calculated from all the values so obtained. This figure corresponds then to the average thickness of the grains in the direction  $Q Q$ . The average thickness of the grains in the direction  $L L$  is obtained analogously by means of the system of parallels to  $L L$ . In the case discussed the results were

in the direction  $Q Q$  6.6 mm.,

in the direction  $L L$  13.5 mm. (lineal magnifying 365 times).  
The grains are therefore twice as long in the direction  $L L$ , as in the direction  $Q Q$ .

If the direction of stretching is not previously known, systems of parallels must be drawn at different angles to  $L L$  and  $Q Q$ . That direction in which the average thickness of the grains is the greatest is the direction of stretching sought for.

In the middle at spot 2 the results were:

Average thickness of grains in direction  $Q Q = 11.1$  mm.

Average thickness of grains in direction  $L L = 11.9$  mm.

Hence it appears that the grains here are not stretched.

On the compressed side at spot 3 we found:

Average thickness of grain in direction  $Q Q = 12.6$  mm.

Average thickness of grain in direction  $L L = 8.3$  mm.

The grains are, therefore, stretched considerably in the direction  $Q Q$ . It is not surprising that the products of the measurements  $Q Q$  and  $L L$  in all three cases do not supply the same figure. As shown by the author in the above-mentioned



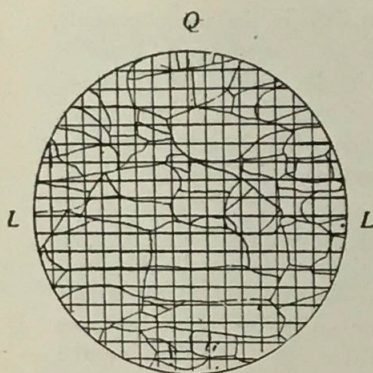


Fig. 10.

Magnified  $\frac{265}{1}$  diameters.

Specimen I. Bent at ordinary temperature. Stretched side. Spot 1.

Average measurements of grains:

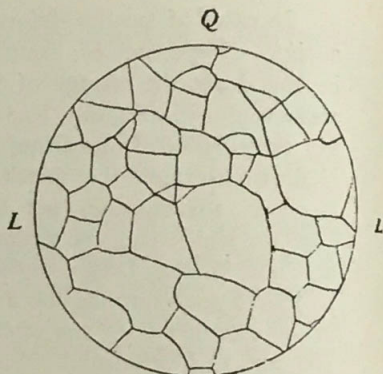
in the direction  $QQ = 6.6$  mm.\*in the direction  $LL = 13.5$  mm.

Fig. 11.

Magnified  $\frac{265}{1}$  diameters.

Specimen I. Bent at ordinary temperature. Middle. Spot 2.

Average measurements of grains:

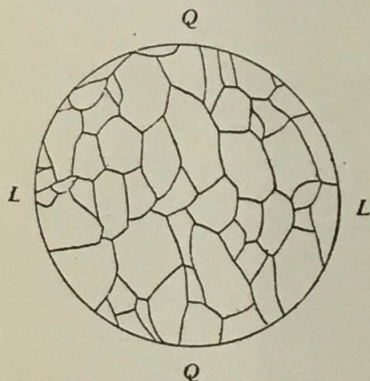
in the direction  $QQ = 11.1$  mm.\*in the direction  $LL = 11.9$  mm.

Fig. 12.

Magnified  $\frac{265}{1}$  diameters.

Specimen I. Bent at ordinary temperature. Compressed side. Spot 3.

Average measurements of grains:

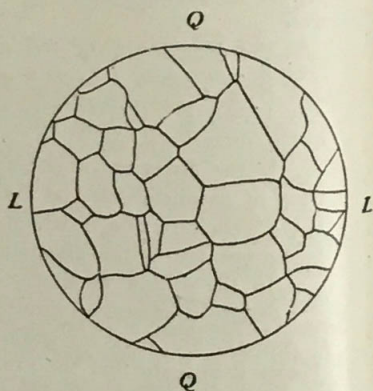
in the direction  $QQ = 12.6$  mm.\*in the direction  $LL = 8.3$  mm.

Fig. 13.

Magnified  $\frac{265}{1}$  diameters.

Specimen II. Bent at blue heat. Stretched side close to the notch.

Average measurements of grains:

in the direction  $QQ = 11.3$  mm.\*in the direction  $LL = 11.4$  mm.

\* Measured under lineal magnifying 365 times.

publication, not only simple changes of form in the ferrite grains take place when there is a permanent deformation in the specimen brought about in an ordinary temperature, but at the same time an increase in the number of grains, that is, a subdivision, occurs.

It is not necessary to mention specially how great is the scope for such researches in testing institutes like the one in Charlottenburg, where problems of very complicated nature have often to be solved.

Specimen II, taken from the same piece of flat iron (see Fig. 8), was heated in an oil-bath to a temperature of  $260^{\circ}\text{C}$ ., and then immediately bent before cooling commenced. The specimen was immediately cracked at the notch, and broke completely, long before the curvature shown in Fig. 9 was attained.

Fig. 13 is a reproduction of a spot close to the notch, magnified  $365$  times. Calculation of the grain measurements by the method described above gave the results

in the direction  $Q Q$  11.3 mm.

in the direction  $L L$  11.4 mm.

Therefore, whereas in the case of Specimen I, bent at an ordinary temperature, considerable stretching of the grains at the corresponding spot in the direction of the bar-axis was determined, there is, in this case, no trace of stretching to be observed. The grains remain completely equiaxial. There is, therefore, the essential difference between cold bendings and bendings at blue heat, that in the former the deformation is distributed over a great mass of material, whereas in the latter, even in the immediate proximity of the fracture, there can be no question of a deformation of the metal.

There was no difference remarked in the microstructure of Specimens I and II at places where there was no deformation. In the case of rupture at blue heat no change of structure occurs, but the iron grains have at blue heat a smaller capacity of deformation than at the ordinary temperature. This is already the second case in which ferrite, without any visible external change, shows really an essentially different behavior with regard to deformation. In the first case the influence of hydrogen was the cause, in the second case the change in its properties produced by blue heat.

I must seize this opportunity to warn against attempts,



unfortunately often enough met with in metallographical literature, which aim without further consideration at drawing conclusions as to the resisting qualities of materials from the state of the microscopical texture, for instance, from the smaller or greater in the manner in which the constitute structural elements are fitted together. This condition of the microstructure, as is known from experience, plays often a very insignificant rôle which, in comparison with other influences, lies far in the background, as is for instance proved by the brittleness produced by blue heat and hydrogen. Metallographical methods can, as will be seen, not be regarded as a universal application, by means of which everything that was formerly problematical can be done and explained without great reflection and expert knowledge. But still they are powerful auxiliaries, which have materially enriched our metallurgical knowledge and promise much for the future, if they are conducted with scientific care and intelligence.

In a second example, to which I should like to direct your attention, it was desired, by means of two copper rods, both of which had been cold-worked to about the same extent, to determine at what minimum temperature the effects of the cold-working disappear. The first object was to determine this degree of temperature from the ratio of the flowing limit to the limit of fracture, which, as is known, increases with the degree of cold-working, and by annealing again becomes nearer to a determinate minimum. Unfortunately, during this experiment the testing material failed, and so it was necessary to employ a metallographical method for arriving at a result.

The following was the method of proceeding. From the material, which was in the form of two flat bars, cross and longitudinal sections were prepared in the state in which the material was delivered and after it had been heated at different temperatures. As shown in Fig. 14 the direction of the shortest side of the specimen is denoted by *a*, the direction of the specimen's axis by *c*, and the direction of the longer side of the cross section by *b*.

In every cross section, therefore, were the directions *a* and *b*, and the longitudinal sections were so made that they contained the directions *a* and *c*. The cut surfaces were polished and etched by means of an ammoniacal solution of the double chloride of copper and ammonia. At different spots on the sections

sketches of the grains were made. Direct photographs are not suitable, because the limits of the grains are only distinctly visible when highly magnified, and in this case the field of vision to be measured is much too small. A method was therefore employed by which first an outline of the sketch was made from a photograph which had been somewhat magnified, and then in this rough sketch the exact course of the grains' boundaries was traced by means of the etching figures visible under high magnifying power.

The measuring of the thickness of the grains in the different directions  $a$ ,  $b$ ,  $c$ , was accomplished by means of netted lines in the manner previously explained. The results are collected in the accompanying tables. The thickness of the grains is referred to the unit  $\mu$  which equals .001 mm. So far as the figures could be ascertained, the results of traction tests are added. In addition the ratio  $\frac{c}{a}$  .100, that is, the ratio of the measurements of the grains in the longitudinal direction and in the direction parallel to the shortest side are given. This ratio, in the case of

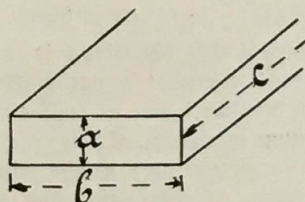


Fig. 14.

equiaxial grains, must correspond approximately to the number 100. On the contrary, if the degree of cold-working be increased, this ratio must also increase. The ratio  $\frac{\sigma S}{\sigma B}$  .100 given in the last column is small in the case of annealed material, and rises with the degree to which the material is cold-worked.

From the tables it can be seen that the quantities  $\frac{c}{a}$  .100 as well as the ratios  $\frac{\sigma S}{\sigma B}$  .100 for both materials in the cold-worked condition, lie very near. The extension of the copper grains is easily perceptible; their measurements in the direction  $a$  are to



those in the direction  $c$  in the proportion of 100:174 and of 100:168 respectively. The limit of flowing (6 S) lies very near to the limit of fracture (6 B), as is generally the case with cold-worked metals. Flat bar I gave already, after being heated 480° C., a value  $\frac{c}{a} \cdot 100$ , which was almost equal to 100; the material is therefore sufficiently annealed; the influence of the cold-working has disappeared; the value  $\frac{\sigma S}{\sigma B} \cdot 100$  has fallen to 12. Heating to a higher temperature has no further effect; the ratio  $\frac{c}{a} \cdot 100$  remains in the proximity of 100.

In the case of material II, however, heating to 480° C. diminished the effects of the cold-working, but did not entirely destroy them.  $\frac{c}{a} \cdot 100$  is still 135, and the value of  $\frac{\sigma S}{\sigma B} \cdot 100$  only lowered from 90 to 74. Heating to 500° C. produced no material further effects; the value  $\frac{c}{a} \cdot 100 = 140$  remained almost unchanged. Heating to 660° C., however, effected the complete annealing of the metal;  $\frac{c}{a} \cdot 100 = 94$  is near the value 100.

It may be remarked that material I is the same copper as that which was already discussed in paragraph 3 (Fig. 7), and which contained the line-like layers of copper-oxidule. The difference of the resisting properties of this copper compared with the flat bar II (poor in oxidule) in the cold-worked state, is evident from the values of contraction and elongation; bar I gives only 16.6 per cent elongation, whereas bar II gives 27.9 per cent. This difference is in accord with the unfavorable condition of copper I during the bending test.

To give an idea of how the copper grains lie, two sketches are given in Figs. 15 and 16 as they were employed for determining the value  $\frac{c}{a} \cdot 700$ . They are lineally magnified 123 times. Fig. 15 is a longitudinal section of the flat bar II after being heated to 660° C. ( $a = 25$ ;  $c = 25 \mu$ . Comp. tables). The grains are equiaxial. Fig. 16 is a longitudinal section of the same material in its original cold-worked state ( $a = 16$  and  $c = 46 \mu$ ). The grains are visibly stretched in the direction  $c$ .

The number of examples in which metallographical methods

## Flat Bar I.

CONDITION	KIND OF SECTION	Average measurements of the copper grains in the direction			$\frac{c}{a} \cdot 100$	Limit of flowing $\sigma S$ kg/squ. mm.	Limit of fracture $\sigma B$ kg/squ. mm.	Contraction	Elongation $\delta = \%$	$\frac{\sigma S}{\sigma B} \cdot 100$
		<i>a</i>	<i>b</i>	<i>c</i>						
		$\mu = 0.001$ mm.								
Cold-worked . . . .	Cross . . . . .	24	27	—						
	" . . . . .	27	30	—						
	Longitudinal	29	—	46						
		26.4*	28.5	46	174	23.5	25.6	34.5	16.6	92
Annealed to 480° C.	Cross . . . . .	28	26	—						
	" . . . . .	24	—	27						
	Longitudinal	26	26	27	104	2.5	21.3	48	48	12
Annealed to 660° C.	Cross . . . . .	33	33	—						
	" . . . . .	33	—	32						
	Longitudinal	33	33	32	97	not determined				

## Flat Bar II.

CONDITION	KIND OF SECTION	Average measurements of the copper grains in the direction			$\frac{c}{a} \cdot 100$	Limit of flowing $\sigma S$ kg/squ. mm.	Limit of fracture $\sigma B$ kg/squ. mm.	Contraction	Elongation $\delta = \%$	$\frac{\sigma S}{\sigma B} \cdot 100$
		<i>a</i>	<i>b</i>	<i>c</i>						
		$\mu = 0.001$ mm.								
Cold-worked . . . .	Cross . . . . .	27	32	—						
	" . . . . .	32	38	—						
	Longitudinal	26	—	46						
		27.4*	35	46	168	22.7	2.48	46.5	27.9	90
Annealed to 480° C.	Cross . . . . .	27	29	—						
	" . . . . .	25	—	35						
	Longitudinal	26	29	35	135	17.7	23.9	45	304	74
Annealed to 500° C.	Longitudinal	22	—	31	140	not determined				
Annealed to 660° C.	Cross . . . . .	28	27	—						
	" . . . . .	25	—	25						
	Longitudinal	26.5	27	25	94	not determined				

\* The most probable values are determined according to E. Vallier (C. r. 128 s. 654-56). To the arithmetical mean ( $m$ ) is added  $-\frac{1}{2} \frac{s_3}{s_2}$  according to the magnitude and sign, where  $s_2$  and  $s_3$  are the algebraical sums of the squares and cubes respectively of the deviations from the mean ( $m$ ) of the single values observed.



have been of service when it was desired to ascertain if the metal had undergone cold-work, and what was the effect and nature of this treatment, is naturally unlimited. I should, however, in conclusion still like to mention one case, viz., that it is possible to determine subsequently, if holes in mild steel were bored or punched, and this can be done even if a part of material be removed from the vicinity of the hole by rubbing or other manipulation. The observations made in this connection are recorded in the Reports of the Royal Technical Testing

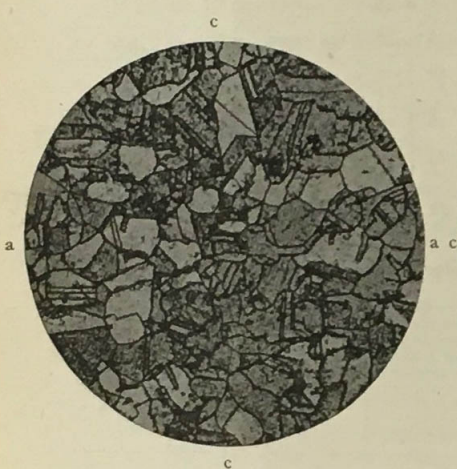


Fig. 15. Magnified 123 diameters. Hand-sketch. Material 11. Heated at  $660^{\circ}$  C. Longitudinal section  $a = 25$ ,  $c = 25$   $\mu$ . Equiaxed copper grains.

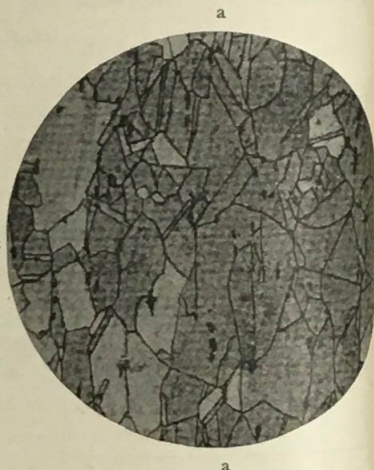


Fig. 16. Magnified 123 diameters. Hand-sketch. Copper material 11. Cold-worked. Longitudinal section  $a = 26$   $\mu$ ,  $c = 46$   $\mu$ . Stretched in direction  $a$ .

Institutes (*Mittheilungen der königlichen Technischen Versuchsanstalten*, 1898, E. Heyn, "Microscopical Researches on Deeply Etched Polished Iron Surfaces"); and further in the Journal of the Society of German Engineers (*Zeitschrift des Vereins Deutscher Ingenieure*, 1900, No. 14, p. 442 ff.).

The microstructure of the material can be compared to hieroglyphics in which is expressed the treatment which the material has undergone.

By deciphering the hieroglyphics valuable information may be obtained. The continual exertions in the field of metallo-

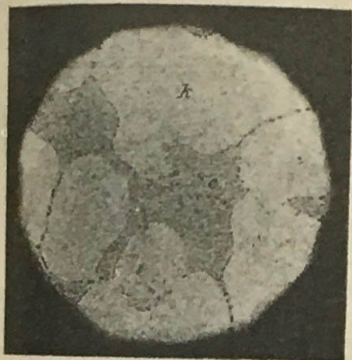


Fig. 17.  $V = 123$ .



Fig. 18.  $V = 123$ .



Fig. 19.  $V = 123$ .



Fig. 20.  $V = 365$ .



Fig. 21.  $V = 123$ .

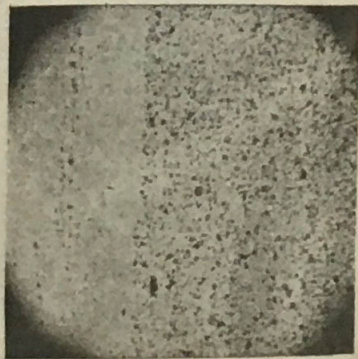


Fig. 22.  $V = 123$ .



graphical research will surely succeed in penetrating the significance of these hieroglyphics, and so afford constantly new and valuable material for the methods of metal testing. Metallographical methods have already established themselves in this branch of experimental science and will continually strengthen their position.

### NICKEL STEEL \*

SINCE the introduction of the Bessemer-Mushet process of steel making whereby the carburization was effected in a fluid state numerous attempts have been made to improve its properties by the introduction of other elements while in the

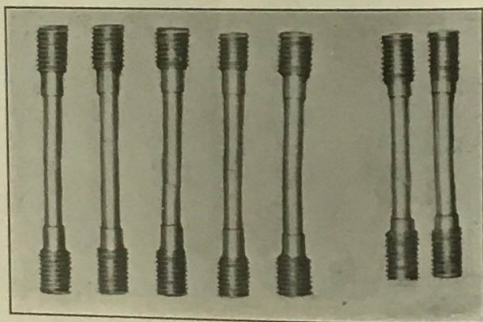


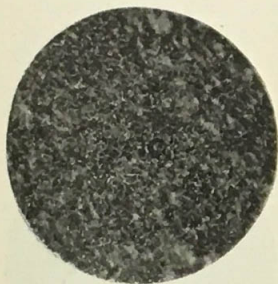
Fig. 1. Test Pieces. 5 Forgings. 2 Castings.

fluid condition. Copper, tin, antimony, manganese, chromium, aluminum, tungsten, nickel, silicon, cobalt, singly or in combination have been tried as alloys with various results, some giving desirable and some very undesirable properties to the alloy. For some years before 1889, Mr. James Riley with Mr. J. F. Hall in England and M. Marbeau in France, experimented with nickel as an alloy with steel and almost simultaneously took out patents for the new material in both countries early in that year. The idea was not new, Sir Henry Bessemer as early as 1858

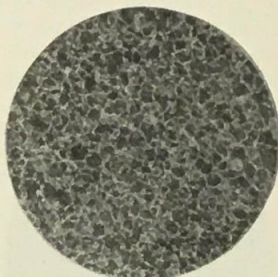
\* From the *Railroad Gazette*, August 8, 1902.

having called attention to the peculiar properties of the ferro-nickel alloys found in meteoric iron, but he himself failed to produce a similar alloy in the crucible furnace.

Mr. Riley, in a paper read before the Iron & Steel Institute some eight months after the granting of the patent, pointed out the advantage to be gained in using a material for all kinds of

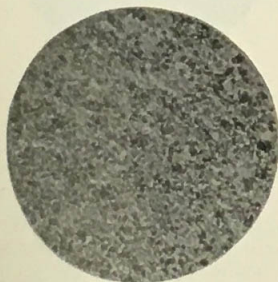


Carbon Steel.

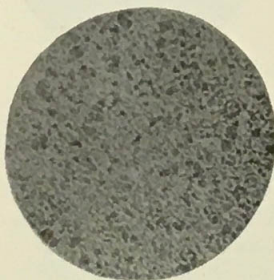


Nickel Steel.

Not Annealed.



Carbon Steel.



Nickel Steel.

Annealed.

Fig. 2.

construction which had an ultimate strength of 30 per cent and an elastic limit of from 60 per cent to 70 per cent greater than mild steel with equal ductility. Since that time the uses of nickel steel have rapidly developed for all kinds of work in which minimum weight with maximum strength is a prime requisite.



The melting point of nickel is  $3,000^{\circ}$  Fahr., slightly higher than that of steel. It is not practicable therefore to add it in the ladle as is done with spiegel or other metals, but it is added with the charge directly, in either the open hearth or crucible methods either in the form of metallic nickel or the oxide, which contains about 77 per cent nickel. Nickel steel is extremely sensitive to sudden changes of temperature, and great care must be exercised to avoid sudden chilling, which may cause surface hardening, making proper working difficult. In general nickel steels, low in carbon, are treated as high carbon steels. The cold metal can be depended upon to resist rough handling and

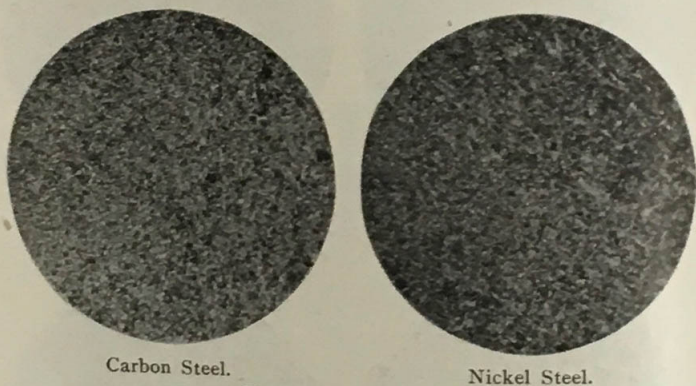


Fig. 3. Oil Tempered.

abuse, but when hot it should be treated with great care. The effect of nickel on low carbon steels or wrought-iron is to form a homogeneous alloy tougher and stronger than either iron or nickel. Low carbon steel cannot be made hard by the addition of nickel alone, which fact has been substantiated by numerous tests. In general the effect of nickel on hardness is not due to the influence of nickel alone, but to its effect on the carbon in rendering that element more sensitive to heat treatment.

Nickel steel is distinguished from simple steel by its high elastic strength. Three per cent nickel alloyed with an open hearth steel of 0.25 per cent carbon produces a metal equal in every way to a simple steel of 0.45 per cent carbon, but having the ductility of low carbon steel. On low carbon steels, not annealed, the addition of each 1 per cent of nickel up to 5 per

cent, causes approximately an increase of 5,000 lbs. elastic limit and 4,000 lbs. ultimate strength. The influence of nickel on the elastic limit and ultimate strength increases with the percentage of carbon present, high carbon steels showing a greater gain than low carbon steels. The addition of nickel raises the proportion of elastic limit to ultimate strength, and adds to the ductility of the steel. Tests made by Mr. A. L. Colby, of the Bethlehem Steel Co., show this ratio to be 46.4 per cent for mild steel, 46.2 per cent for medium hard steel, and 58.7 per cent for medium hard nickel steel. This effect of nickel upon the elastic limit accounts for the increased working capacity of nickel steel and its resistance to molecular fatigue.

This particularly valuable alloy is steadily growing in use for locomotive parts, and for some time it has been in general favor with marine engine builders on account of its increased strength, which permits a great saving in weight. Some of the uses to which it is being put in locomotive building are given below.

#### *Nickel-Steel Forgings*

Driving-wheel axles.  
Tender truck axles.  
Piston-rods.  
Connecting-rods.  
Guides.  
Crank or wrist pins.  
Guide yoke (bearer).  
Spring links.

#### *Nickel-Steel Castings.*

Frames and rails.  
Wheel centers.  
Driving-boxes.  
Crossheads.  
Saddles (cellars).  
Rocker-shaft.  
Steam-chests.  
Guide yoke knees.  
Eccentric straps.  
Equalizing beams.  
Reverse shaft.  
Cross-ties.  
Cylinder heads.  
Furnace bearers.  
Water-space frame.  
Drawhead-pocket.  
Lifting-link.

It will be seen that this list includes most of the heavy parts of the engine together with those subjected to the greatest stress.

The Bethlehem Steel Co., who were among the first to make nickel steel in this country, and who have had much to do with its successful 'introduction into the arts, recently furnished the



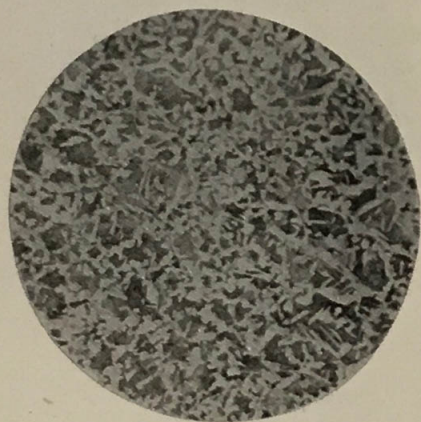
nickel steel forgings and castings for a narrow-gauge locomotive which showed the following excellent physical properties determined from standard  $2\frac{1}{2}$  in.  $\times$   $\frac{1}{2}$  in. tensile specimens and 1 in.  $\times$   $\frac{1}{2}$  in. bending test bars.

*Nickel-Steel Forgings.*

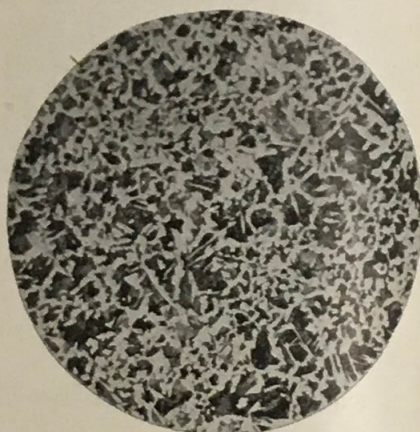
	Tensile strength, lbs.	Elastic limit, lbs.	Exten. Per cent	Cont. Per cent
Driving-wheel axles . . . . .	90,310	64,170	25.00	53.76
Piston rods . . . . .	90,140	60,090	25.50	54.08
Main crank-pins . . . . .	93,570	65,450	24.00	49.37
Front crank-pins . . . . .	92,180	64,170	24.50	51.00
Connecting-rods and guides . . . .	92,040	59,820	26.00	53.01

*Nickel-Steel Castings.*

Crosshead . . . . .	84,549	53,980	18.50	31.10
Furnace-bearer, bearer guide . . .	85,050	54,490	18.00	26.04

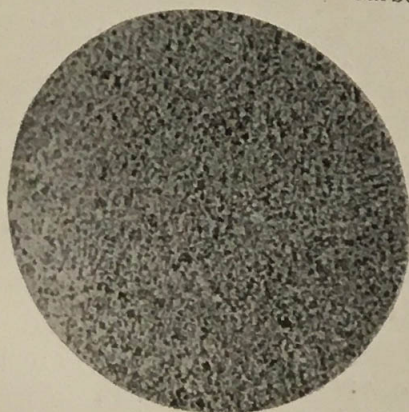


Edge.

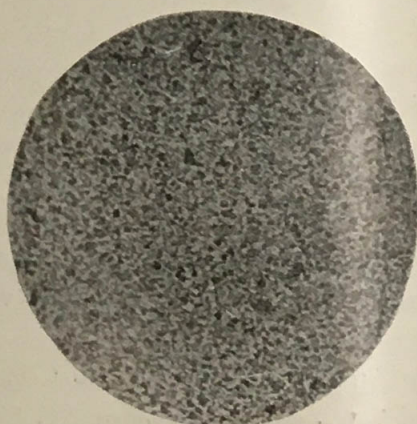


Center.

Carbon Steel.



Edge.



Center.

Fig. 4. Nickel Steel.

Fig. 1 shows the appearance of the test pieces after fracture. Assuming \$.03 per lb. as the cost of introducing nickel into the forgings and castings for this locomotive, it cost only about \$70 to very materially increase the life of the working parts, and most of this extra cost is returned to the purchaser when he returns this nickel steel to the steel maker as scrap.

Photo-micrographs of nickel and carbon steel under the same heat treatment, Figs. 2, 3 and 4, show the uniformly fine crystalline structure of nickel steel as compared to carbon steels.

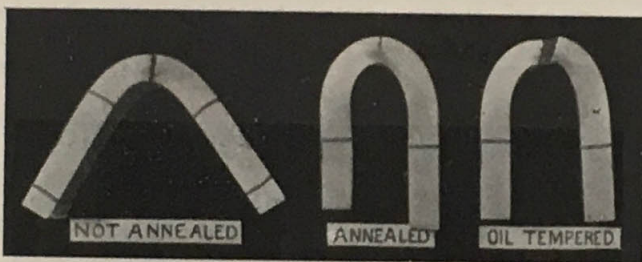


Fig. 5. Carbon Steel Bending Test Pieces.

Fig. 4 shows the difference between the structure on the inside and outside of an axle, those taken from the outside portion illustrating the hardening effect of finishing shown by the finer and more homogeneous nature of the crystals.

The following table gives the physical properties in a comparative manner of nickel and carbon steels under different heat treatments, and Figs. 5 and 6 give a comparison of the ductility of the two metals under bending tests.

	Tensile strength, lbs.	Elastic limit, lbs.	Exten. Per cent	Cont. Per cent
Annealed:				
Carbon steel . . . . .	109,500	51,440	19.50	36.31
Nickel steel . . . . .	100,330	66,720	25.00	54.56
Oil-tempered:				
Carbon steel . . . . .	129,360	67,230	17.50	338.5
Nickel steel . . . . .	103,890	76,390	25.00	61.56

The wonderful results that can be obtained from nickel steel forgings by special heat treatment are shown in the table below.



Note the unusually high elastic limits obtained without sacrifice of either extension or contraction of area.

*Small Rifle Barrels — Nickel Steel.*

Tensile strength, lbs.	Elastic limit, lbs.	Exten. in 2 in. Per cent	Cont. of area. Per cent
115,100	99,820	23	64.00
114,080	97,780	23	64.95
114,590	99,820	23	65.45
116,620	96,770	22.50	62.05
116,120	97,780	23	64.00
114,590	98,800	24	62.53

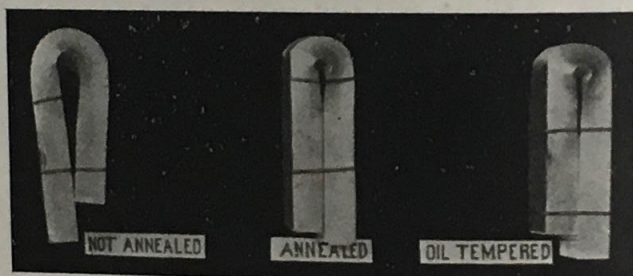


Fig. 6. Nickel Steel Bending Test Pieces.

With the present increased facilities for its manufacture on a large scale, its high elastic limit, together with its ductility as compared to other materials of construction, nickel steel is slowly coming into general use despite its added first cost. In no other field does it hold out so many advantages to be gained by its adoption as in locomotive building. Here great strength must be had, and the problem is to keep the weight of the parts within such limits that the additional boiler power required may be supplied without increased total weight. Its ability to resist sudden shocks, its ductility and high elastic and ultimate strength give it a great advantage over carbon steels, wrought or cast iron. Its place in marine engineering is now assured, and its introduction into new fields will be watched with interest.

## PHOTO-MICROSCOPY OF METALS AS PRACTICED BY STEEL COMPANIES.\*

By M. A. RICHARDS

WHEN Henry Clifton Sorby of England reported the results of his study of the structure of meteoric and artificial irons at a meeting of the Iron and Steel Institute in 1864, in a paper "On the Microscopical Structure of Meteors and Meteoric Iron," very little attention was paid his report. After a lapse of twenty-two years the Institute requested Dr. Sorby, Dr. Percy, and Sir Henry Bessemer to decide what was the best way of illustrating a complete paper on the microstructure of iron and steel. As the result of this investigation, Dr. Sorby presented two papers to the Iron and Steel Institute "On the Microscopical Structure of Iron and Steel." Since that time this means for the examination of metals and their alloys has increased in importance and value, and within the last decade has become a recognized department in the testing laboratories of many industries, especially those of producers and users of iron and steel.

An equipment for the study of metal-microscopy or metallography consists of: a means for the preparation of the micro-section; reagents to "etch" the specimen; a compound microscope, which, in addition to the usual accessories, is equipped with a vertically movable stage and a means for "vertical illumination"; a light, strong, steady, and of uniform intensity, and a camera adapted to use with a microscope.

The piece of steel to be examined and photographed, having been machined to a suitable size for use on the microscope stage (the most convenient size and shape being either cylinders or cubes of about one-half inch), the surface to be examined is filed to a true plane, and the polish is continued on successively finer grades of emery-cloth stretched over plane surfaces. It is well to change the direction of the application of the abrasive

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\* *Journal of Applied Microscopy*, August, 1902. The accompanying illustrations are reproduced here through the courtesy of the *Journal of Applied Microscopy*.



with each change in fineness, so that each set of scratches is at right angles with the previous set of marks. One set of marks must be entirely obliterated by the following set, and care must be taken to keep the polished face a plane surface or else difficulty will be met with in getting a flat field under the microscope. Following the polish on emery-cloth the process is continued on sheets of muslin or canvas also stretched over plane surfaces and to which is applied the best Turkish flour-of-emery. Then follows a polish upon a similarly covered disk coated with a paste of calcined ammoniacal alum and powdered castile soap dissolved in water. Finally, the effacement of all scratches is completed upon a chamois skin stretched over a plane iron disk and coated with the best jeweler's rouge mixed with water. A properly polished specimen of steel, upon the completion of this process, should be microscopically free from all scratches and should look like a well-burnished piece of nickel or silver plating.

Where a large number of specimens are to be prepared, the hand-process described is too slow, and a polishing "head" is used, driven by power and so arranged that four surfaces are at the command of the polisher. Generally these four surfaces are an emery-wheel or a carborundum disk of considerable fineness; a disk covered with canvas or linen duck for use with flour-of-emery or carborundum powder; a third disk covered with felt or billiard-cloth upon which is used alum and castile soap paste, and a fourth face for the chamois skin and rouge polish.

The polishing powders are most conveniently applied by means of bristle brushes to the revolving disks in the form of pastes made up with water. The application should be frequent rather than in greater quantities at longer intervals. For the sake of cleanliness the water supply arranged to trickle constantly on the disks of most grinding machines should be abandoned and the pastes applied wet enough not to need additional water.

To bring out the micro-structure of iron or steel it is generally necessary to apply an "etching" solution, although this is not always needed. Fig. 1 is a micro-photograph of a section of pig iron as it appears at the completion of the polishing process; no etching having been done. This is styled polishing

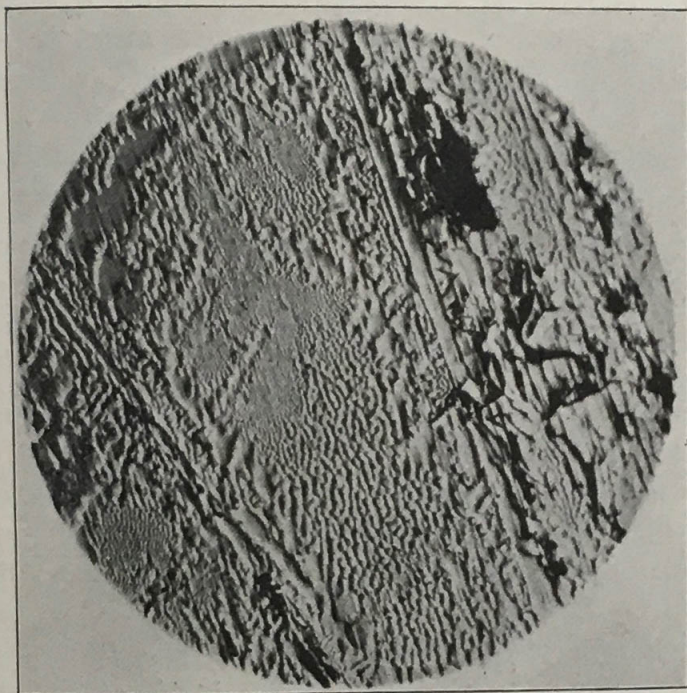


Fig. 1. Pig Iron polished in relief.

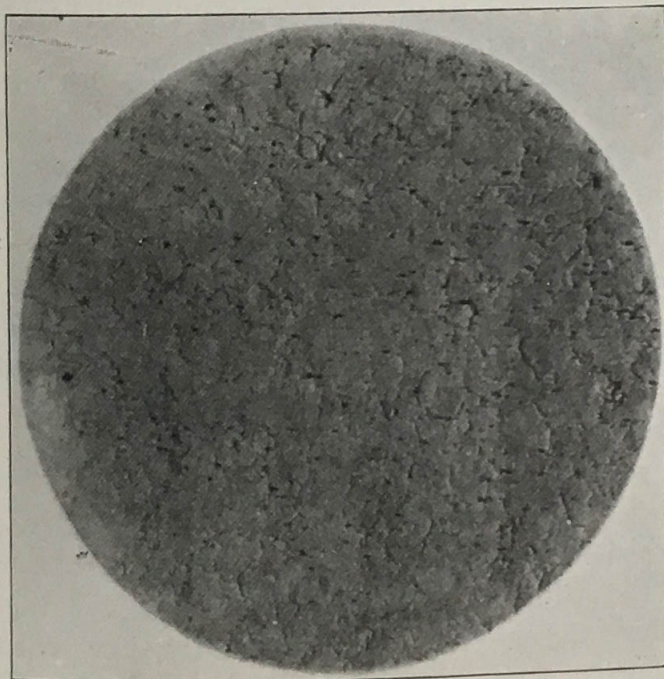


Fig. 2. Steel after polishing.



in "relief." Fig. 2 is a micro-photograph of a piece of steel before etching, and Fig. 3 is the same piece after the application of a 25-per cent solution of tincture of iodine in alcohol as an "etch."

The reagents used for etching iron and steel are various, depending mainly on the carbon-content of the steel and its heat-treatment. For mild and untreated steel, tincture of iodine, full-strength, and in a 25-per cent solution in alcohol, is used; for hardened steels 5-per cent, 10-per cent and 20-per cent solutions of nitric acid in alcohol are very satisfactory.

The polished surface of the micro-section should be freed from all grease and dirt by washing in alcohol and carefully dried. If the etching fluid is iodine, it is best applied with the tip of the finger or a camel's-hair brush, keeping finger or brush lightly rubbing over the surface being etched and carefully watching the process, to prevent too deep an etching. The time necessary to etch properly a specimen cannot be definitely stated, as it varies greatly, ranging from a few seconds to a minute, depending upon the nature of the steel and the etching solution employed. A beginner will find it best to err on the safe side of stopping the process before the etching is complete; for should the examination show the structure insufficiently developed, the etching may be repeated until a desired result is obtained. A rule (with exceptions, of course) is to stop the action of the etch when specular appearance of the specimen has just disappeared and before the surface becomes dull and uniformly gray.

Having decided that the section is sufficiently etched, quickly hold the specimen under a tap of flowing water for a few moments, then remove all traces of moisture by the application of a few drops of alcohol, and finally dry it in a blast of air or by careful friction with very clean, old, and washed-out linen or cotton cloth.

Diluted nitric acid is used as an etching medium in case of hardened steel. With increase in hardness and working there is an increase in the fineness of the micro-structure of steel, to develop which it is best to use the more dilute solution of nitric acid. At the same time, the finer structures are the more difficult to develop, and take a much shorter time to etch; hence it is particularly true that several short applications of the etch are more likely to produce the result sought for than one pro-



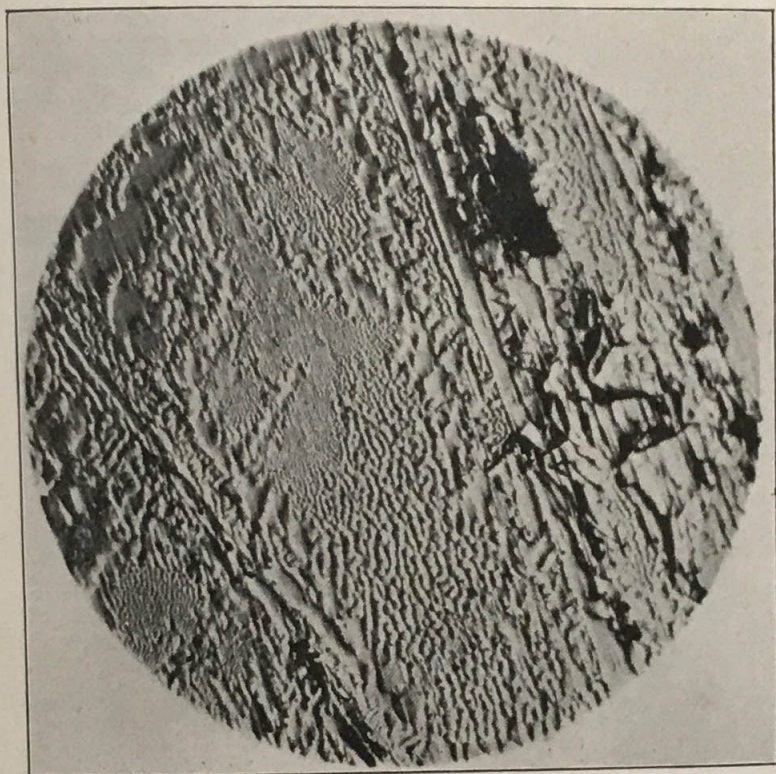


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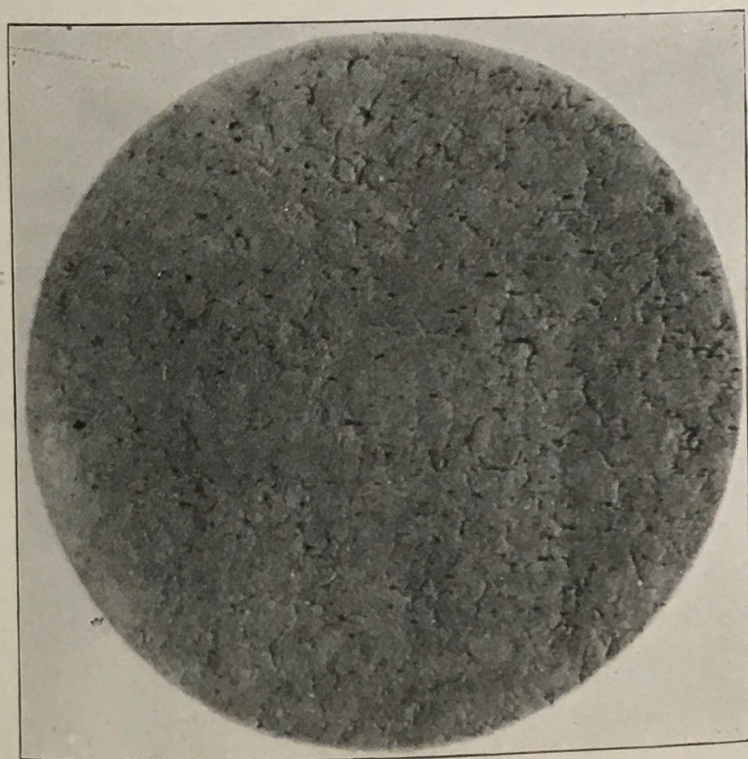


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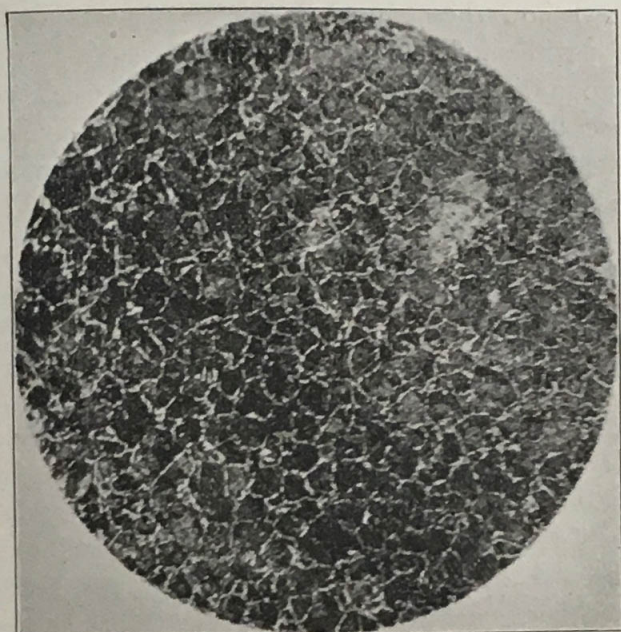


Fig. 3. Same as Fig. 2 after polishing.

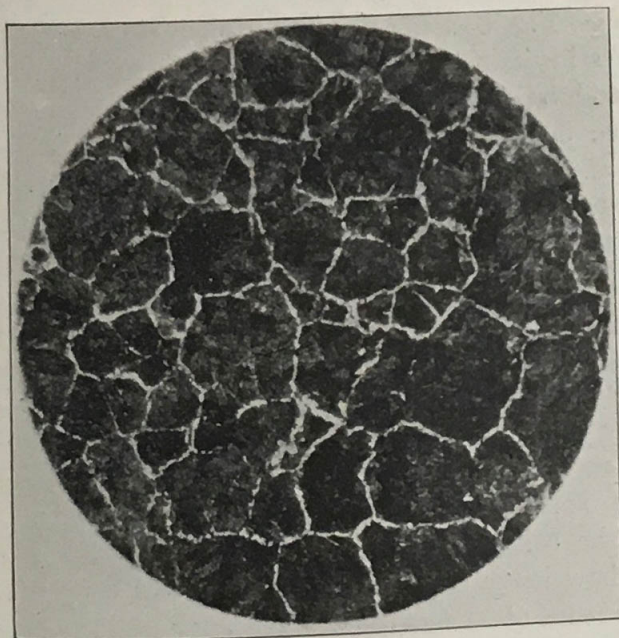


Fig. 4. Steel heated to 1200° C.



longed etching would. Examination between successive applications shows how the etching is progressing.

To etch with nitric acid, pour a small quantity of the proper dilution into a suitable dish, and, holding the cleaned micro-section, face down, with pincers or a straight-nosed crucible tongs, dip the face to be etched at least twice into the etching fluid, immediately remove and let the action continue with face of section turned up, so that the action may be watched. When it is complete (two seconds is often enough), hold in stream of water and dry as described before. If examination shows etching to be incomplete repeat the process; if too deeply etched, repeat the polishing.

Various devices for temporarily mounting specimens, especially for those of irregular shape, are supplied by dealers, but none has been found more convenient than a small one and one-half inch square of ground glass upon which is a lump of beeswax the size of a hazel nut. It need not necessarily be ground glass, but generally an identification-mark is needed, and then the ground surface is very convenient to write on.

To mount an irregular section so that the etched surface is parallel to the ground-glass base, a microtome is a great convenience (*M*, Fig. 8). Projecting from a cylindrical metal base three inches in diameter, is a threaded upright three inches high and one and one-half inch in diameter. A cylindrical nut or collar three inches high and two and one-half inches outside diameter screws on the threaded upright. A small circle of chamois skin is placed on the top of the threaded upright to protect the etched face of the micro-section. To mount a section, place it face down on the chamois skin, press upon the upper projecting portion the lump of beeswax and upon this place the ground glass (ground surface down). A few revolutions of the collar will cause the glass to rest upon the upper edge of the collar, and the adhesion of the glass and beeswax to the specimen may be made complete by slowly turning the collar down with one hand while keeping the glass base in close contact with the collar-top with the other hand. In this manner, no matter how irregular the section, the parallelism of the etched surface and the glass base may very quickly and accurately be obtained.

The magnification used in ordinary investigations ranges

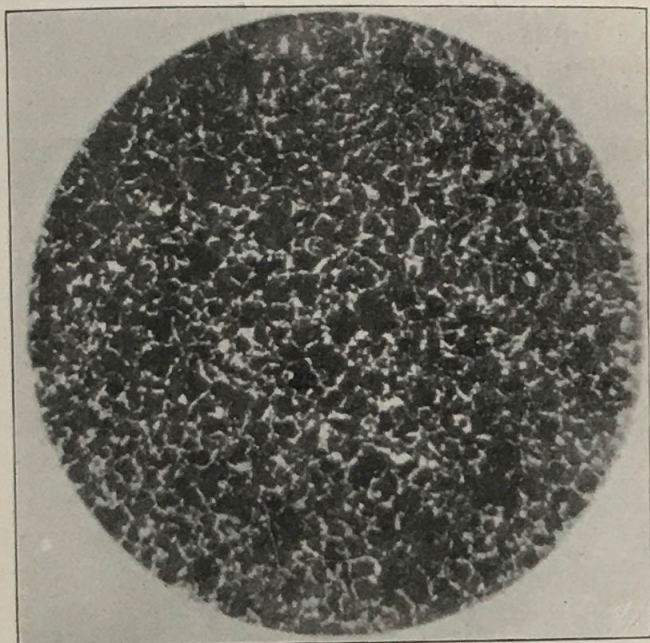


Fig. 5. Same steel as Fig. 4 heated to 630° C.

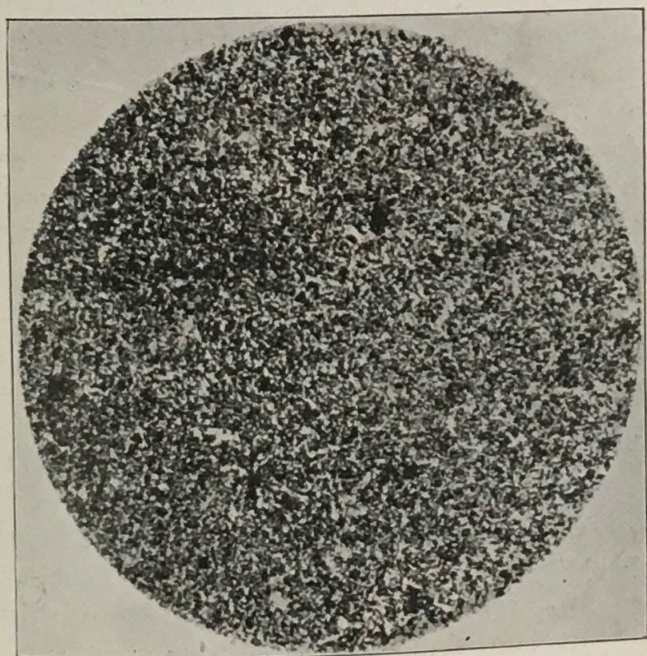


Fig. 6.



from 30 to 100 diameters. The micrographs accompanying this article are uniformly 65 diameters. Photo-micrographs 4 and 5 are from the same bar of medium carbon steel etched with tincture of iodine for ten seconds. The difference in the size of the grains of the two specimens is caused by the degree of heat to which the two sections were subjected; 4 having been heated to 1200° C. and 5 to 630° C.

When the examination shows that the specimen has been properly prepared, the observer selects a spot representative of the micro-section under consideration. Here the temptation arises to take that which is unusual rather than that which is characteristic, or to photograph "freaks" instead of normal structures. Unusual structures frequently occur in metals or alloys that have been subjected to a variety of treatments in the way of reheating or reforging. Figs. 6 and 7 were taken from the same micro-section of steel within a circle of less than one-half inch radius, and show a variety sometimes found in a single specimen.

Arranged upon a well-equipped optical bench, in the following order, are these parts (see Fig. 8): a source of light, the best (after sunlight) being an automatic-feed arc lamp (*A*), surrounded by a suitable case, with an opening at one side, in front of which is arranged a system of condensing lenses (*B*), similar to those used in high-grade projection lanterns; a cell filled with a saturated solution of alum (*C*), to absorb the heat rays; a double-convex lens (*D*), to further concentrate the beam of light; a holder for color and ground-glass screens (*E*); an iris diaphragm and a photographic shutter (*F*) with pneumatic release; and probably another double convex lens (*G*), all of which are so secured to the optical bench as to be movable in a horizontal direction for the purpose of focusing the light on the vertical illuminator. All the members of this system are also adjustable vertically, so they may be brought into alignment with the source of light.

The beam of light leaving the lens system enters the body of the microscope (*H*, whose tube is vertical), through the opening of the vertical illuminator, inserted between the objective and the main tube. The vertical illuminator contains either a plain glass disk reflector or a total reflection prism, which changes the direction of the horizontal beam of light to a perpendicularly descending beam. The objective of the microscope

acts still further as a condenser and an exceedingly well-illuminated spot is thus secured on an opaque body.

The camera used in the photomicrography of metals is generally one adapted to  $4 \times 5$  inch plates, and is so arranged that it may be swung out of the observer's way while an examination is made. The cut shows it in this position. When the part to

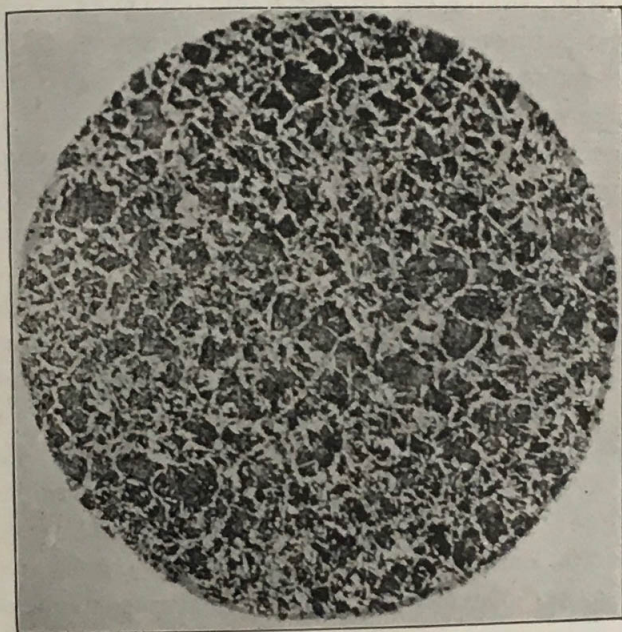


Fig. 7.

I            H            G            F            E            D            C            B            A

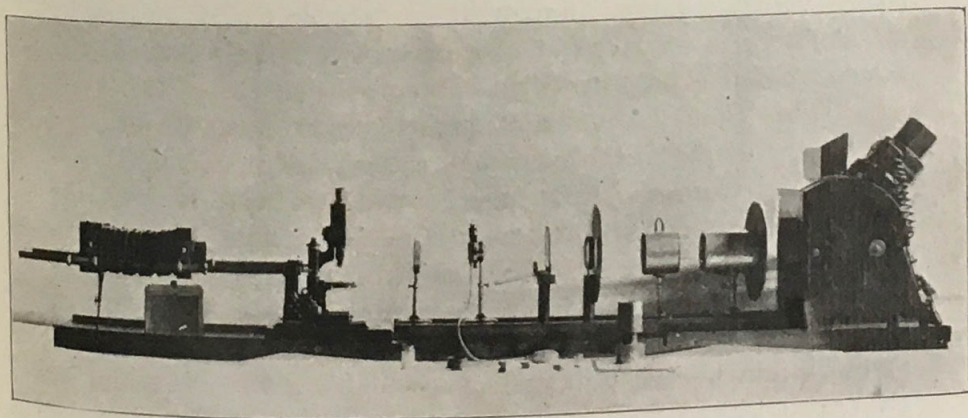


Fig. 8. Optical Bench for Photo-microscopy of metals.



be photographed is selected, the eye-piece of the microscope is removed, the collar (*K*) to connect the microscope and the camera is placed over the end of the tube, and the eye-piece is replaced within the collar. The camera is swung into position vertically over the microscope, the front board of the camera is lowered until its collar telescopes with that on the microscope. The upper, or ground-glass, end of the camera is moved up or down until the circle of the projected image is the size desired. The ground glass of the camera has cemented to its center a large cover-glass, giving a clear-glass spot in the middle of the image. The final focusing for sharpness and clearness of the micro-image is done with a focusing eye-piece (*J*) set on the clear spot in the ground glass, and is obtained by the use of the slow-motion screw of the microscope.

Slow isochromatic plates have proven the most satisfactory for all-around use, as they give the color values without the use of a color screen, thus avoiding long exposures, with their attendant ills, vibration of the optical bench and irregularity in illumination.

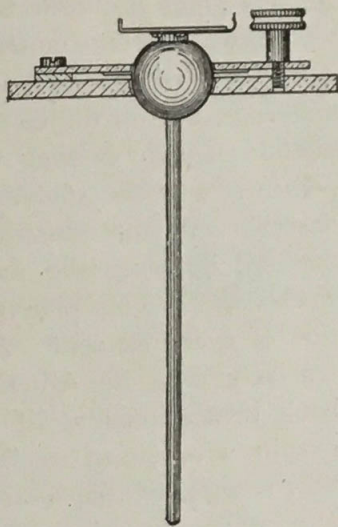
With an electric arc light of about 2000 candle power, a system of good condensers, a prism reflector, and using a magnification of 65 diameters, an exposure need not be longer than twelve seconds. Developed with ordinary pyro developer, a negative giving good, contrasty prints should be obtained. A glossy paper should be used to reproduce a micro-negative.

If it is desired to preserve the micro-section it should be coated with a solution of paraffin in benzole and placed under a bell jar or in an air-tight case containing a vessel filled with calcium chloride or other hygroscopic substance.

To be of practical value a complete record should be kept of the history of the metal examined, embracing its chemical composition, its treatment with regard to heating and forging, the results of the physical tests, nature of the etching solution employed, magnification and illumination used, and any other related facts that may be considered of importance. Special pains should be taken clearly to state the location of the micro-section in the original piece of metal and whether the surface photographed is longitudinal or transverse. Card index files are published, which make possible the combination of the mounted micro-photograph with all of the data mentioned.

## METALLOGRAPHIC NOTES

**A New Tilting Stage.** — The tilting stage devised by Prof. A. K. Huntington, and which was mentioned in the last issue of *The Metallographist* (page 341), is clearly shown in the accompanying illustration. It consists of a pair of brass plates with two circular openings. If the microscope has a stage which can be racked up and down, the end of the stem attached to the ball can very readily be brought down against a roughened plate, and the movements of the mechanical stage then used for levelling the object. On the upper portion of this ball is attached a small stage to take the specimen mounted roughly on a one-inch glass plate, and from the lower portion of which projects a long rod, by which the specimen is orientated. A clamp screw to adjust the pressure on the ball is provided. The tilting stage is placed on the microscope stage, and the specimen having been mounted roughly on glass plate one inch wide and placed in the holder, may be adjusted till the surface to be examined is exactly at right angles to the optic axis. Where the microscope upon which it is used has a mechanical stage, an adjustable plate may be placed at the lower portion of the microscope, so that it may be forced against the lower end of the long rod, thus temporarily fixing it. When this is done, the ordinary movements of the mechanical stage form a fine adjustment for levelling the object. When the object is levelled, the plate which holds the rod attached to the ball may be released, and the mechanical stage movements used in the usual manner for moving the object.



**Recent Publications.** — *The Analysis of Steel-Works Materials*, by Harry Brearley and Fred Ibbotson. 500 pages. Pub-



lished by Longmans, Green & Co., London. — The book is divided into thirteen parts. Part I deals with the analysis of steel, and includes the determination of carbon, silicon, manganese, sulphur, phosphorus, chromium, nickel, tungsten, molybdenum, vanadium, titanium, aluminum, copper, arsenic, iron and some minor constituents. Part II is devoted to the analysis of pig iron, and Part III to the analysis of steel-making alloys, including the analysis of iron alloys containing silicon, manganese, chromium, nickel, tungsten, molybdenum, vanadium, titanium, aluminum and boron. In Part IV some rapid furnace analyses are described. Part V is devoted to the analysis of ores; Part VI, to the analysis of refractory materials; Part VII, to the analysis of slags; Part VIII, to that of fuel; Part IX, to the analysis of boiler water, boiler scale, etc., and Part X, to the analysis of engineering alloys. Part XI is devoted to the micrographic analysis of steel; Part XII, to pyrometry, while in Part XIII some miscellaneous notes are collected.

It was not the author's intention to be exhaustive in their description of the methods used for the analysis of steel-works materials, but on the contrary, to confine their attention to those methods which, in their opinions, are the most accurate and expedient. For the analysis of sulphur in steel, for instance, they describe only the classical gravimetric method and one evolution method, ignoring the many other volumetric methods used in steel-works. This plan is followed in describing the determination of every element. Many methods, therefore, which are extensively used by American chemists are not described in the book, if mentioned at all. Twenty pages are devoted to the micrographic analysis of steel. The authors write as follows: "It is now recognized that a steel may actually break down in use, even although its chemical analysis and mechanical tests are perfectly satisfactory, so that in such cases the microscopic method of examination becomes a most invaluable accessory. At the same time it is perhaps advisable to point out that the indications of microscopy should not be accepted as final; complete knowledge of chemical constitution, mechanical tests and heat treatment, must be correlated with that of the ultimate structure as revealed microscopically." The methods used for the preparations and examinations of the samples in the metallographic laboratory of the Sheffield University College are described to the exclusion

of other methods. The authors espouse Prof. J. O. Arnold's views on metallography, and treat the subject accordingly. Drawings and photo-micrographs of wrought iron and of various grades of steel, forged, cast, annealed and hardened, are reproduced and described. The chapter on pyrometry is confined to a description of the Le Chatelier thermo-electric pyrometer.

A most exhaustive bibliography of steel-works analysis, compiled by Harry Brearley, is appended to the book, and should prove of much value and interest to chemists. It fills no less than 135 pages and includes 1858 titles of articles accompanied by short outlines of the subject treated. The labor of preparing such admirable bibliography must have been very great, and the author is entitled to the gratitude of all those interested in the analysis of steel-works materials.

The typography, illustrations, paper and general getting up of the book are excellent. On the whole, this work should be of considerable value and interest to the chemist.

*Assaying and Metallurgical Analysis*, by E. L. Rhead and A. Humboldt Sexton. 431 pages. Published by Longmans, Green & Co., London. — The purpose of this book and the treatment of the subject which it covers are described by the authors in a preface which we reproduce in part: "The object which the authors had in view in writing the present work was to provide the student, chemist, or assayer with a handbook sufficiently comprehensive to include the greater part of the work likely to be required in the laboratory or assay office. Most of the approved methods in general use are included; but it would be impossible, even if advisable, to include all the variations which have been suggested by competent workers.

"The direction for work and the description of processes have been made as concise as possible, to reduce the size of the book; but by the inclusion of the chemical reactions of the metal it has been sought to make the reasons for the procedure clear, and where it was thought necessary, chemical explanations have been given at greater length. Care has been taken to make the methods of calculation clear, using for the most part the approximations of the atomic weights commonly employed. In a few instances directions are given for conducting experiments to ascertain the kind of treatment suitable for a given ore, and the examination of works, products and solutions has also been described.



"Part I is devoted to laboratory appliances and general processes, useful hints rather than full description being given.

"In Part II the general plan followed has been to consider under the name of each metal, (1) the materials and products in which its estimation is commonly required, giving a brief description of their characters so as to make their identification easy; (2) the dry tests for, and important chemical reactions of the metal, with, in some cases, special instructions for the detection of small quantities; (3) the methods of conducting the dry assay by fusion or otherwise, followed by application to special materials where modifications are necessary; (4) methods of dissolving substances and preparing solutions for the determination of the metal; (5) methods for the gravimetric, volumetric, and calorimetric determination, with the precautions necessary.

"In Part III — metallurgical analysis — an attempt has been made to group the very various substances to be dealt with in a compact manner, and, by cross-references inserted in the text, to, as far as possible, avoid repetition. The use of all specially marked measuring apparatus has been avoided, and the preparation of standard and other solutions required for a particular assay has been incorporated with the description of the method."

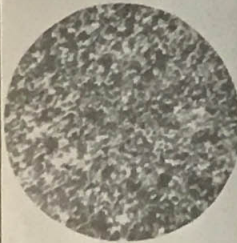
This book is undoubtedly a valuable addition to our literature on the analysis of metallurgical products.

**Errata.** — In the last issue of *The Metallographist* (p. 341) mention was made of a recent lawsuit in which metallography played an important part. Through a clerical error it was stated that the judgment was "reversed," while it should have read "reserved." The judgment has now been rendered, and on the technical points involved the judge decided in favor of the defendants, stating that the failure of the rod was due to the weakness of the design, and not to defective material. The plaintiffs, however, won the case on a legal point arising out of the wording of the insurance policy.

On page 268 of the October, 1902, issue of this journal, Slide No. 5 should read Slide No. 4, and vice versa. Slide No. 5 was magnified 58 diameters, and not 85, as indicated. On page 285, Slide No. 33 represents Spiegeleisen, and not Silicospiegel.

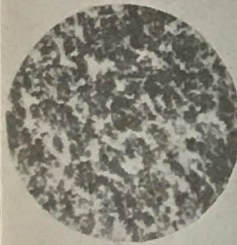






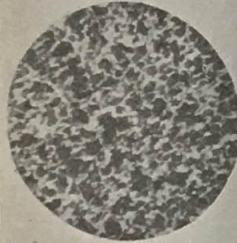
93

STEEL UNTREATED  
TS = 81250 LBS. PER SQ. INCH  
EL = 43410  
%C = 0.285 %C = 46.5



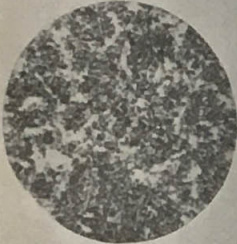
94

HEATED TO 1000°C AIR COOLED  
TS = 80850 LBS. PER SQ. INCH  
EL = 41070  
%C = 0.285 %C = 45.3



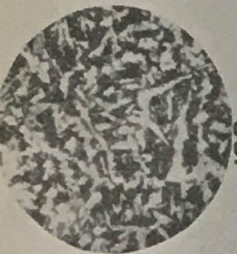
95

HEATED TO 875°C AIR COOLED  
TS = 51000  
EL = 41850  
%C = 0.27 %C = 42.6



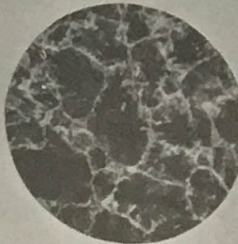
96

HEATED TO 750°C AIR COOLED  
TS = 89000  
EL = 43310  
%C = 0.26 %C = 44.5



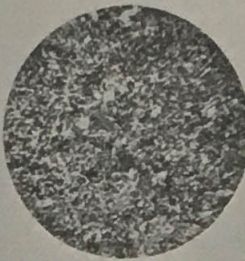
100

HEATED TO 750°C FURNACE COOLED  
TS = 78850  
EL = 43310  
%C = 0.26 %C = 50.3



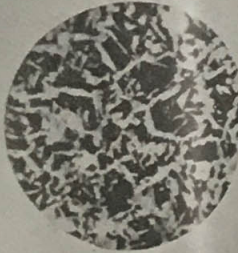
104

HEATED TO 1000°C FOR ONE HOUR AIR COOLED  
TS = 55700  
EL = 46400  
%C = 0.22 %C = 43.5



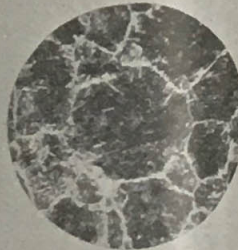
103

HEATED TO 750°C FOR ONE HOUR AIR COOLED  
TS = 90850  
EL = 48250  
%C = 0.30 %C = 46.3



102

HEATED TO 1000°C FURNACE COOLED



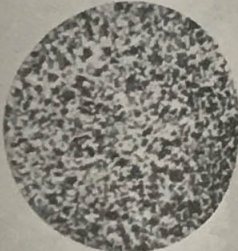
99

HEATED TO 1000°C AIR COOLED



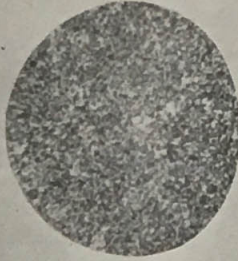
98

HEATED TO 1000°C AIR COOLED



101

HEATED TO 800°C FURNACE COOLED



97

HEATED TO 750°C FURNACE COOLED

# EXPERIMENTS SHOWING THE EFFECT OF HEAT ON THE PHYSICAL PROPERTIES AND STRUCTURE OF STEEL 33% CARBON THE WESTINGHOUSE MACHINE CO. PITTSBURGH, PA.

# The Metallographist

A QUARTERLY PUBLICATION DEVOTED TO THE STUDY OF METALS, WITH  
SPECIAL REFERENCE TO THEIR PHYSICS AND MICROSTRUCTURE,  
THEIR INDUSTRIAL TREATMENT AND APPLICATIONS.

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## THE CONSTITUTION AND THERMAL TREATMENT OF STEEL

By H. M. HOWE

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CONSTITUTION. — The great advance which has taken place in our knowledge of the constitution of steel and the other varieties of iron has shown that they resemble very closely the igneous and metamorphic rocks; i.e., exactly those which, like the different varieties of iron, have formed from the cooling of molten or at least pasty masses. Just as a granite on close examination is seen to consist of an aggregation of crystalline fragments of mica, quartz and feldspar, each of which is a perfectly definite chemical compound, with definite crystalline form and definite physical properties in general so the microscope shows us that a given piece of steel or iron usually consists of extremely minute crystalline particles of two or more substances, each of which is a definite entity, with definite chemical composition and definite physical properties, but besides the granitic type certain varieties of iron seem to represent the obsidian type. In this, as in aqueous solutions, the ratios in which the different chemical substances, the silica, lime, etc., exist are not fixed or definite; they vary



from case to case; not *per saltum*, as between definite chemical compounds, but by infinitesimal gradations. The different substances present appear to be dissolved as it were in each other in a sort of solid solution which presents the indefiniteness of composition, the incapacity of being resolved by any magnification of the microscope, and the feeble chemical attractions between the different components, characteristic of a solution. The schistose structure of rock masses, their columnar or basaltic structure arranged in columns perpendicular to the cooling surface, their "vugs" or cavities lined with specimens of free crystals, their segregation, etc., are reproduced in a most interesting way in metallic masses.

Of these different microscopic entities which constitute the different varieties of iron, only the following here need consideration:—

1. *Ferrite*, the name assigned to the microscopic particles of nearly—perhaps perfectly—pure metallic iron. It is very soft and ductile.

2. *Cementite*, a definite carbide of iron,  $\text{Fe}_3\text{C}$  very brittle, harder than hardened steel, scratching glass and feldspar, but not quartz ( $H = 6$ ), and present in proportions which, in a general way increase with the proportion of carbon present. It is the substance to which chilled cast iron usually owes its hardness and brittleness.

3. *Pearlite*. Slowly cooled steels in general consist essentially of a mixture of ferrite and cementite, in proportions corresponding to the carbon-content of the mass, as a whole. But these two substances habitually interstratify as a "eutectic"\* conglomerate called pearlite in the ratio of about seven parts of ferrite to one of cementite, hence containing about 0.90 per cent carbon. Steels containing either more or less carbon than 0.90

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\* A eutectic is the last freezing part of an alloy and corresponds to what the mother-liquor of a saline solution would become if such a solution, after the excess of saline matter was crystallized out, were finally completely frozen. It is the mother-liquor of "Bittern" frozen. Its striking characteristics are: (1) that for given metals alloyed together its composition is fixed, and does not vary with the proportions in which those metals are present, the "excess-metal" freezing before the eutectic; (2) that though thus constant, its composition is not in simple atomic proportions; (3) that its freezing point is constant; and (4) that it consists of interstratified layers of the metals which compose it. See Alloys and Fusion.

per cent, that is, an excess of cementite or ferrite over the eutectic ratio of 7:1 are a conglomerate of pearlite plus this excess. They are called hyper-eutectic or hypo-eutectic according as this excess is cementite or ferrite.; i.e., according as their carbon-content is above or below the 0.90 per cent, which the eutectic itself contains.

4. *Martensite*, the characteristic and chief constituent of hardened steel is a hard brittle mass with a needle-like structure consisting of iron containing carbon in proportions which vary from nothing up to about 2 per cent. As it has not yet been resolved by the microscope into different components, we regard it provisionally as a "solid solution" the ultimate constituents of which are iron and carbon; as to the nature of its proximate constituents we can, as yet, only speculate. When it contains 0.90 per cent of carbon and thus corresponds in ultimate composition to pearlite, it is called *hardenite*.

5. *Graphite*, a characteristic component of "gray cast iron" of which it usually forms from 2.50 to 3.50 per cent. It is nearly pure carbon in very thin laminated plates or flakes, often curved. In gray cast iron these flakes form a nearly continuous skeleton; and as they readily split open when a piece of this iron is broken, rupture passes through them with the result that even though the graphite may form only some 3 per cent of the mass by weight (say 10 per cent by volume) practically nothing but graphite is seen in the fracture, hence the weakness and the dark gray fracture of "gray cast iron," and hence by brushing this fracture with a wire brush and so detaching these loosely adherent flakes of graphite, the color can be changed nearly to the very light gray of pure iron.

6. *Slag*, the characteristic component of wrought iron, in which it is usually present to the extent of 0.20 to 2 per cent, is essentially a silicate of iron, and is present in wrought iron simply because this product is made by welding together pasty granules of iron in a bath of such slag, without ever melting the resultant mass or otherwise giving the envelopes of slag thus imprisoned a chance to escape completely.

7. *Austenite*, *troostite*, *sorbite*, and other constituents have also been described.

8. Fig. 1 shows how the occurrence of these constituents varies both with the temperature and with the proportion of car-



bon present; the Roman letters indicate the constituents normal and stable in each of the many fields into which it is divided. Before considering this diagram we must recognize the further complication in the constitution of iron, due to its having at least three distinct allotropic modifications, *Alpha*, *Beta*, and *Gamma*, each corresponding to a distinct range of temperature.

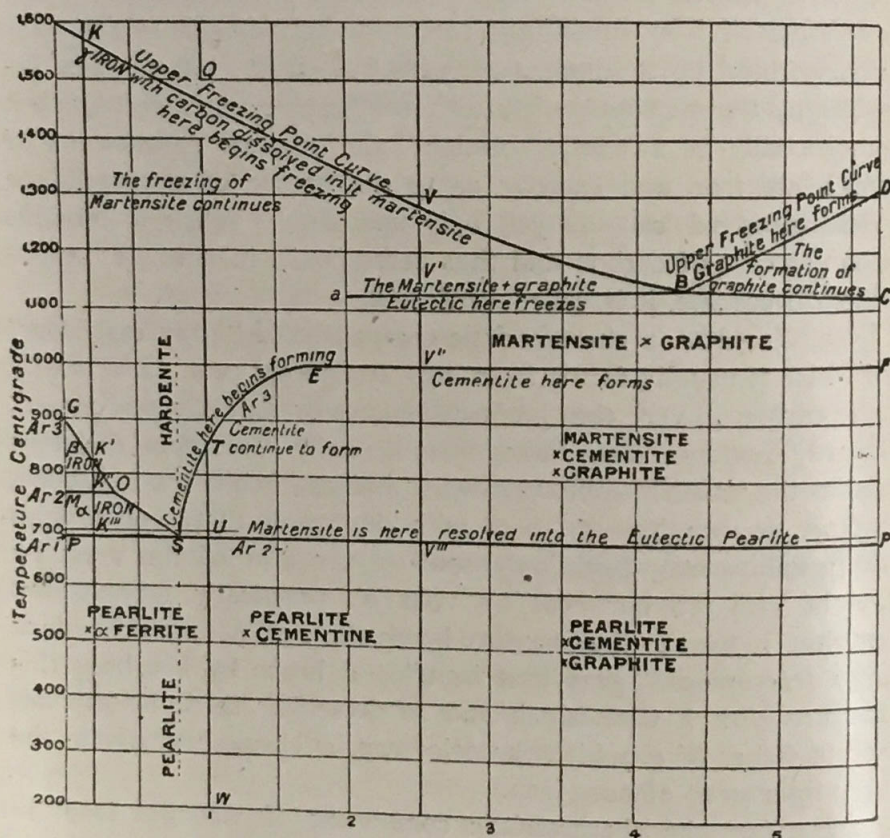


Fig. 1.

9. *Alpha*-iron is the weak, ductile, magnetic variety, stable below  $A_2$  Fig. 1, characteristic of wrought iron and of low carbon steel.

N.B. The "carbon-content of this diagram refers only to the carbon actually combined with the iron and excludes that which is present as graphite. This may be regarded as a foreign body. For instance, a gray cast iron containing 3.50 per cent of

carbon of which 3 per cent is graphite and only 0.50 per cent combined, may be looked upon as in effect a hypo-eutectic steel, approximately rail steel, containing 0.50 per cent of carbon, but contaminated with 3 per cent of graphite as a foreign body.

10. *Beta-iron* is the non-magnetic variety stable between  $A_2$  and  $A_3$ , probably very hard and brittle and probably characteristic of certain self-hardening steels, such as manganese steel containing 7 per cent of manganese and of normal or "carbon" steel when hardened by sudden cooling.

*Gamma-iron* is the non-magnetic variety stable above  $A_3$  characteristic of nickel and twelve per cent manganese steels and probably relatively hard but ductile.

In Fig. 1,  $AB$  and  $BD$  represent the freezing point of iron for different proportions of carbon and the lines below these,  $aBc$ ,  $GOS$ ,  $SE$ ,  $MO$ , and  $PSP$  represent spontaneous retardations which occur during the undisturbed further slow cooling, showing that at these several lines heat is evolved within the metal and hence that some molecular rearrangement occurs. Each of the tripartite groups of lines, (1)  $AB$ ,  $BD$ ,  $aBc$  and (2)  $GOS$ ,  $SE$ ,  $PSP$ , is of the family of curves of the solidification of those solutions which in freezing break up to form a mechanical mixture of (a) a eutectic and (b) the component of that eutectic which is in excess over the eutectic ratio. The natural inference that these two groups here represent analogous occurrences is thus far well supported by the evidence.

To illustrate the meaning of the diagram let us follow by means of the ordinate  $QUW$  the undisturbed slow cooling from the molten state of a hyper-eutectic steel containing 1 per cent of carbon. When the gradually falling temperature reaches  $1460^\circ \text{C.}$  ( $Q$ ) the mass as a whole freezes gradually, forming martensite, apparently simply a solidified solution of carbon in *Gamma-iron*, which, however, preserves the essential characteristics of a solution. The heat evolved during solidification retards the fall of temperature: after this the rate of cooling remains regular until  $T$  ( $850^\circ$ ) on the line  $STE$  ( $Ar_3$ ) is reached, when a second retardation occurs due to the heat liberated by the passage within the pasty mass of part of iron and carbon from a state of mere solution to that of definite combination in the ratio  $\text{Fe}_3\text{C}$  forming microscopic particles of cementite while the remainder of the carbon and iron continue dissolved in each



other as martensite. This formation of cementite continues as the temperature falls, till at about  $690^{\circ}$  C. ( $U$ , called  $Ar_{2-1}$ ) so much of the carbon (in this case about 0.10 per cent) and of the iron have united in the ratio of cementite, that the composition of the remaining solid solution or "mother-metal" of martensite has reached that of hardenite, i.e., it now contains 0.90 per cent of carbon. As the temperature now falls past  $690^{\circ}$  this hardenite mother-metal in turn splits up after the fashion of eutectics, into alternate layers of ferrite and cementite grouped together as pearlite, so that the mass as a whole now becomes a mixture of pearlite with cementite. The iron thus liberated as the ferrite of this pearlite, changes simultaneously to *Alpha*-ferrite. The passage of this large quantity of carbon and iron, 0.90 per cent of the former and 12.60 of the latter, from a state of mere solution as hardenite to one of definite chemical union as cementite, together with the passage of the iron itself from the *Gamma*-state to the *Alpha*-state, evolves so much heat as actually to heat the mass up so that it brightens in a striking manner. This phenomenon is called "recalcescence." The change from martensite to ferrite and cementite is accompanied by the loss of the hardening power, as will be explained shortly.

To take a second case, molten hypo-eutectic steel of 0.20 per cent of carbon on freezing at  $K$  passes to the state of martensite, *Gamma*-iron with this 0.20 per cent of carbon dissolved in it. Its further cooling undergoes three spontaneous retardations, one is at  $K'$  ( $Ar_3$ ) at about  $820^{\circ}$  C., at which part of the iron isolates itself in separate crystals within the solid solution as ferrite, and in crystallizing passes from the *Gamma*-state to the *Beta*-state, that is free iron of the *Beta* allotropic modification. At the second retardation  $K''$  ( $Ar_2$ ), which occurs at about  $770^{\circ}$  C., this ferrite changes to the normal and magnetic *Alpha*-state, so that the metal becomes magnetic. Moreover the crystallization of ferrite within the martensite which began at  $Ar_3$ , continues until  $690$  degrees or  $Ar_1$  is reached, by which time so much iron has separated out that the remaining mother-metal has reached the composition of hardenite; i.e., it now contains 0.90 per cent of carbon. Again, as the temperature in turn falls past  $Ar_1$ , this hardenite mother-metal splits up into cementite and ferrite grouped together as pearlite, with simultaneous change of this ferrite to the *Alpha*-state and with the resulting recalcescence. All these phenomena are



parallel with those of 1 per cent carbon steel at this same critical point,  $Ar_1$ . As such steel cools slowly past  $Ar_3$ ,  $Ar_2$ , and  $Ar_1$ , it loses its hardening power progressively. In short from  $Ar_3$  to  $Ar_1$  the excess substance, ferrite or cementite, hypo- and hyper-eutectic steels respectively, progressively crystallizes out within the martensite mother-metal which thus progressively approaches the composition of hardenite, reaching it at  $Ar_1$ , and there splitting up into ferrite and cementite interstratified as pearlite. Further, any ferrite liberated at  $Ar_3$  changes there from *Gamma* to *Beta*, and any present at  $Ar_2$  changes from *Beta* to *Alpha*. Between *O* and *S*,  $Ar_3$  and  $Ar_2$  occur together, as do  $Ar_2$  and  $Ar_1$  between *S* and *P'* and  $Ar_3$  and  $Ar_2$  and  $Ar_1$  at *S* itself; so that these critical points in these special cases are called  $Ar_{3-2}$ ,  $Ar_{2-1}$  and  $Ar_{3-2-1}$  respectively. The corresponding critical points which occur during rise of temperature, with the reverse transformations, are called  $Ac_1$ ,  $Ac_2$ ,  $Ac_3$ , etc. *A* (Chernoff) being their generic name, *r* referring to falling temperature (*refroidissant*), and *c* to rising temperature (*chauffant*, Osmond).

To take a third case, that of hyper-eutectic steel containing 2.50 per cent of carbon, and thus approaching the limits of cast iron: since some 2 per cent of carbon suffices to saturate solid *Gamma*-iron, the martensite which progressively freezes out as the temperature sinks below *V*, tends to reject, and thus to leave in the still molten matrix of entangled mother-metal, all carbon in excess of this quantity. This progressive separation of martensite with consequent enrichment of the mother-metal in carbon, continues with further fall of temperature until, when  $1125^\circ \text{C.}$  (*V'* on *aBc*) has been reached, the carbon-content of the mother-metal has reached 4.30 per cent, which is that of the martensite plus graphite, eutectic. As the temperature now sinks past *aBc*, this mother-metal in turn freezes as a whole, splitting up into the eutectic conglomerate of martensite plus graphite, and, as in the other cases, the heat thus liberated retards the cooling. When the temperature falls to 1,000 degrees, part of the graphite already formed unites with part of the iron of the martensite to form cementite; so that between *V''* and *V'''* the mass is a conglomerate of martensite, cementite and graphite. On reaching  $Ar_1$  at *V'''*, this martensite (now become hardenite) as usual splits up into pearlite and the mass now becomes a conglomerate of pearlite, cementite, and graphite, and remains so after cooling is complete.



As we pass to cases with higher and higher carbon-content, the martensite which begins freezing on cooling past the line  $AB$  forms a smaller and smaller proportion of the whole, and the martensite-graphite eutectic which forms at the second freezing point,  $aB$ , increases in amount until, when the carbon-content reaches 4.30 per cent there is but a single freezing point, and the whole mass, when solid is made up of this eutectic. If there be more than 4.30 per cent of carbon, then not martensite but graphite solidified between  $BD$  and  $Bc$ , but, as before, the martensite-graphite eutectic freezes at the lower freezing point  $Bc$ . But in either case the changes occur at  $EF$  and  $PSP'$ , which have been described by hyper-eutectic steel of 2.50 per cent of carbon.

Just as the presence of carbon shifts the position of the freezing point and of the various critical points, so does that of other elements, notably silicon, phosphorus, manganese, chromium, nickel, and tungsten. Nickel and manganese lower these critical points, so that, with 25 per cent of nickel or with 13 per cent of manganese,  $Ar_3$  lies below the common temperature of twenty degrees C. These steels then normally consist of *Gamma*-iron, modified by the large amount of nickel or manganese with which it is alloyed. They naturally are non-magnetic, or very feebly magnetic. But the critical points of such nickel steel, though thus depressed, are not destroyed; and if it be cooled in liquid air below its  $Ar_2$ , it passes to the *Alpha* state and becomes magnetic.

**THERMAL TREATMENT.**—The hardening, tempering, and annealing of steel, the chilling and annealing of cast iron, and the annealing of malleable cast iron, are readily explained by the facts just set forth. Sudden cooling hardens and embrittles steel and cast iron because it retains the high-temperature martensite state, by denying the time needed for these successive transformations which, in slow cooling, finally yield a mixture of pearlite, either with ferrite or with cementite alone, or with cementite and graphite; in the cold these transformations cannot take place, thanks to molecular rigidity or other cause. The suddenly cooled metal is hard and brittle because the cold martensite which it contains is hard and brittle. The degree of hardening which steel undergoes increases with its carbon-content; either (1) because the hardness of martensite increases with its own carbon content, or (2) because during sudden cooling the presence of carbon, like a brake, impedes the change from martensite to pearlite with ferrite



or cementite, thus leading to a more complete retention of the martensite, or of the intermediate *Beta*-state of the ferrite; or (3) for both reasons.

But these transformations, though retained by the sudden cooling, still tend to occur; if we reheat hardened steel to, say,  $250^{\circ}\text{C}$ . ( $482^{\circ}\text{F}$ .) as in tempering, we so far relax this restraint that part of the martensite, thus forcibly retained, changes to pearlite with ferrite or cementite. We undo the effect of the sudden cooling, to a degree which increases with the temperature of reheating or "tempering," and with the degrees of molecular freedom which it gives. If the reheating be carried still farther, to a dull red heat, the restraint is wholly relaxed, so that the change from martensite to pearlite and cementite becomes complete; the metal is as soft and ductile as if the cooling had been slow instead of sudden; it is annealed.

"Chilling" cast iron, i.e., hastening its cooling by casting it in a cold mould, restrains the separation of graphite by hurrying the metal through the range between  $ABD$  and  $EF$  in which graphite tends to form, and retains in its stead the supersaturated martensite which the metal forms on sudden solidification, more or less split up into pearlite and cementite on passing  $Ar_{2-1}$ . The annealing of such iron may occur in either of two degrees: a small one, as in making common chilled cast iron objects, such as railway car wheels, or a great one, as in making malleable cast iron. In the former case the objects are heated only to the neighborhood of  $Ar_1$ , so that the martensite may resolve itself into pearlite and cementite. The joint effect of such chilling and such annealing is to make the metal much harder than if slowly cooled, because for each 1 per cent of graphite which the chilling suppresses, the annealing substitutes 15\* per cent of the glass-hard cementite.

In making "malleable cast iron" the re-heating is carried to a temperature near or perhaps above  $EF$ , so that the graphite-forming tendency, suppressed by the initial relatively rapid cooling — a suppression favored by the special composition of the cast iron used — asserts itself. Since graphite and iron combine to form cementite when the temperature sinks past 1,000 degrees, re-heating the metal above 1,000 degrees should cause cementite

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\* Fifteen per cent, if we add together both the "excess" cementite and that contained in the eutectic pearlite, which result finally from the carbon which has been prevented from separating as graphite.



to split up into martensite plus graphite. Graphite now forms slowly within the mass; but whereas the graphite which separates when common cast iron objects cool slowly from the melting point forms large flakes which weaken and embrittle the metal by breaking up its continuity, that which now separates forms a fine powder, naturally much less injurious. For this heating the castings are packed in a mass of iron oxide, which at this temperature gradually removes the fine or "temper" graphite by oxidizing that in the outer crust to carbonic oxide, whereon the carbon farther in begins diffusing outwards by "molecular migration" to be itself oxidized on reaching the crust. This removal of graphite doubtless further stimulates the formation of graphite by relieving the mechanical and perhaps the osmotic pressure. Thus, first, for the brittle glass-hard cementite and martensite there is gradually substituted the relatively harmless temper graphite; and secondly, even this is in large part removed by surface oxidation.

Each of these ancient processes thus consists essentially in so manipulating the temperature that, out of the several possible constituents, the metal shall actually consist of a special set in special proportions. But in addition there is another very important principle underlying many of our thermal processes, namely that the state of aggregation of certain of these constituents, and through it the properties of the metal, as a whole, are profoundly affected by temperature manipulations. Thus, prior exposure to a temperature materially above  $A_{c1}$  coarsens the structure of most steel, in the sense of giving it, when cold, a coarse fracture, and enlarging the grains of pearlite, etc., found in the slowly cooled metal. This coarsening and the brittleness which accompanies it increase with the temperature to which the metal has been exposed. Steel which after a slow cooling from about  $722^{\circ}\text{C.}$  will bend  $166^{\circ}$  before breaking, will, after slow cooling from  $1,050^{\circ}\text{C.}$  bend only  $18^{\circ}$  before breaking. This injury fortunately can be cured either by *re-heating* the steel to  $A_{c1}$ , when it "refines," i.e., returns spontaneously to its fine-grained ductile state (*cooling* past  $A_1$  does not have this effect); or by breaking up the coarse grains by *mechanical distortion*, e.g., by forging or rolling. For instance, if steel has been coarsened by heating to  $1,400^{\circ}\text{C.}$ , and if, when it has cooled to a lower temperature, say  $850^{\circ}\text{C.}$ , we forge it, its grain-size and ductility when cold will be approximately those which it would have had if heated only to  $850^{\circ}$ . Hence steel which



has been heated very highly, whether for welding, or for greatly softening it so that it can be rolled to the desired shape with but little expenditure of power, ought later to be refined, either by re-heating it from below  $A_{r_1}$  to  $A_{r_1}$ , or by rolling it after it has cooled to a relatively low temperature, i.e., by having a low "finishing temperature." Steel castings initially have the extremely coarse structure due to cooling without mechanical distortion from their very high temperature of solidification; they are annealed, i.e., this coarseness and consequent brittleness are removed, by re-heating them to the critical range, which also relieves the internal stresses due to the different rates at which different layers cool, and hence contract during and after solidification. For steel containing less than some 0.13 per cent of carbon the embrittling temperature is in a different range, near  $700^{\circ}\text{C.}$ , and such steel refines at temperatures above  $900^{\circ}\text{C.}$

When we consider the great number of different regions in Fig. 1, each with its own set of constituents, and remember that by different rates of cooling from different temperatures we can retain in the cold metal these different sets of constituents in widely varying proportions; and when we further reflect that not only the proportion of each constituent present but also its state of aggregation can be controlled by thermal treatment, we see how vast a field is here opened, how great a variety of different properties can be induced in any individual piece of steel, how enormous the variety of properties thus attainable in the different varieties collectively, especially since for each percentage of carbon an incalculable number of varieties of steel may be made by alloying it with different proportions of such elements as nickel, chromium, etc.

As yet there has been only the roughest survey of certain limited areas in this great field, the further exploration of which will enormously increase the usefulness of this wonderful metal.



## THE FRACTURE OF METALS UNDER REPEATED ALTERNATIONS OF STRESS \*

By J. A. EWING and J. C. W. HUMFREY

IT is well known that metals will break down after repeated application and especially under repeated reversal, of stresses greatly less than those that have to be applied when the ultimate strength of the material is tested in the ordinary way. The researches of Wohler have shown, for example, that iron capable of bearing about 20 tons per square inch of steady load will break when it is exposed to some millions of reversals of a stress of 8 or 9 tons per square inch alternately in compression and extension. When the alternating stress is increased, a smaller number of reversals sufficed to produce rupture. On the other hand examples such as are furnished in the balance spring of a watch, or in a railway axle, show that very many millions repetitions may be applied with impunity, provided the limit of greatest stress be kept sufficiently low. The mild steel axle of a railway carriage is exposed to many millions reversals of a stress, which, in some cases, approaches a value as high as in the rare instances where fracture of such axles has occurred, an explanation is to be found in the gradual spreading of a crack from an origin supplied by an air-bubble or other primitive defect in the material. But Wohler's researches, which have been confirmed by other observers, give evidence that a stress not much greater than this, and far below not only the ultimate strength but even the "yield-point" of the metal, will produce what is called "fatigue" and bring about fracture when reversal of the stress is repeated many times.

The purpose of this paper is to describe experiments in which the microscope has been applied to study the nature of the process of fatigue by which breakdown occurs under repeated reversals of stress. The experiments have been made during the past year in the Engineering Laboratory at Cambridge. The metal chosen for experiment was Swedish iron, of high and very uniform quality. It had the further advantage for our purpose of pos-

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\* Philosophical Transactions of the Royal Society of London. Read November 20, 1902.



sessing a clearly defined and fairly large crystalline structure, well adapted when polished and etched to exhibit the characteristic lines known as "slip-lines" or "slip-bands," which appear in ordinary testing when any portion of the material has passed its limit of elasticity under strain.\* We used the metal in the form of rods with a rectangular section, the dimensions being approximately 0.3 inch by 0.1 inch, and to make the structure as uniform as possible these were in all cases annealed by being kept for about

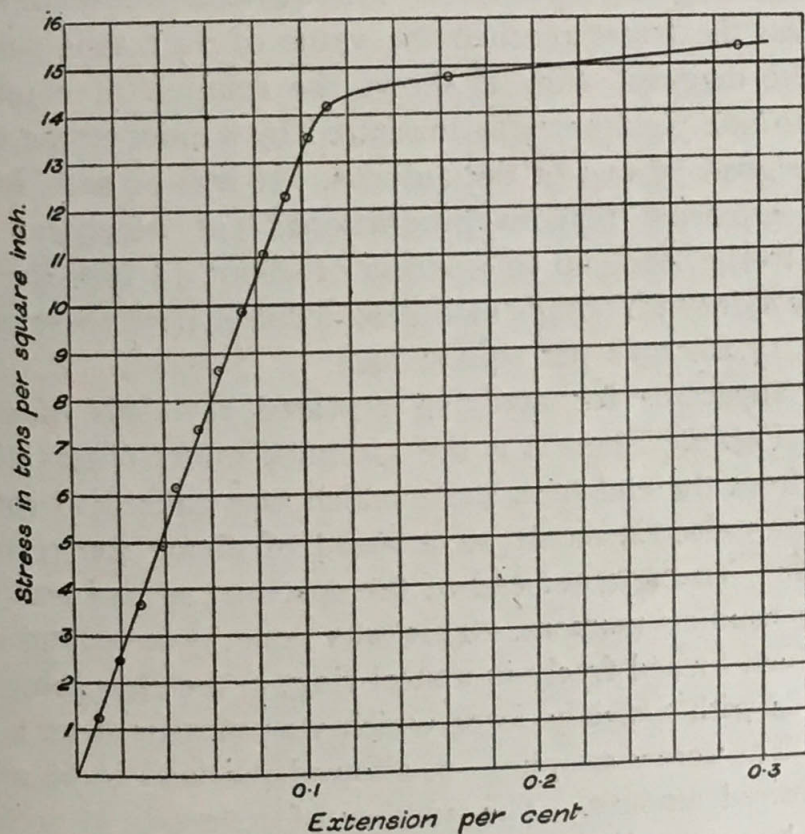


Fig. 1.

two hours at a dull heat, while enclosed in a tube filled with lime, in a muffle furnace. One of the surfaces of each rod was polished and etched, and the rod was subjected to reversals of stress by bending, so that the polished surface was alternately extended and compressed. This was done, as in Wöhler's original experiments, by making the rod project from a revolving shaft with a load on the projecting end. As the process went on the rod was from time to time examined under the microscope, and in

\* Ewing and Rosenhain, "The Crystalline Structure of Metals," Bakerian Lecture, 1899, 'Phil. Trans.,' A, vol. 193, p. 353. — Also *The Metallographist*, vol. III, 1900, p. 94.



several cases photographs of the same crystals were taken at each stage to record the progressive effect of repeated reversals of stress.

A tensile test of the Swedish iron rod used in these experiments, carried out in a testing machine in the usual manner, showed a breaking strength of 23.6 tons per square inch (reckoned on the original area of section), with an ultimate extension of 0.8 inch in a length of 3 inches, and a contraction of area at the break amounting to 61 per cent. There was a well-marked yield-point when the stress reached the value of 14.1 tons per square inch. The diagram, Fig. 1, shows the relation of extension to load up to this yield-point as measured by a microscope extensometer designed by one of the authors. It will be seen from this that the extension remains proportional (as nearly as can be judged) to the load, up to a stress of about 13 tons per square inch. The value of Young's modulus, deduced from these measurements, is 13,200 tons per square inch.

The apparatus for applying repeated reversals of stress is shown in Fig. 2. There *a* is the specimen under observation. It projects from the end of a shaft which was caused to rotate, by means of an electric motor, at a speed of about 400 revolutions per minute. To the outer end of the specimen a load was applied causing a bending moment. This was done by attaching a brass cap, *b*, which turned freely in a steel ring, *c*, the ring being pulled downwards with a steady force which was measured on a spring balance. The screw coupling, *d*, allowed the load to be adjusted to any desired amount. The number of reversals of stress was recorded by a revolution counter, *e*. The specimen under test was filed to a uniform rectangular section of about 0.3 inch by 0.1 inch. Part of one of the broad faces was polished and was, in general, etched by dilute nitric acid. The specimen was inserted in the grip at the end of the shaft, adjusted to run true, and the desired load was applied. After making a certain number of reversals the specimen was taken out for microscopic examination; it was then replaced for a further run, and so on, no difficulty being experienced in replacing it each time in the same position as at first.

Before the experiments were made it had been conjectured that the destructive effect of repeated alternations of stress might be ascribed to a loosening of the intercrystalline cement rather



than to damage of individual crystals. Previous experiments had shown that in fracture by ordinary progressively augmented strain the material gives way, in general, not at boundaries, but through the crystals themselves,\* but it seemed possible that the effect of repeated straining might be different in this respect. By way of testing the point the experiment was made of subjecting an unnotched specimen to many reversals of stress, in order to see whether the intercrystalline boundaries became apparent as they would do if yielding took place between each crystal and

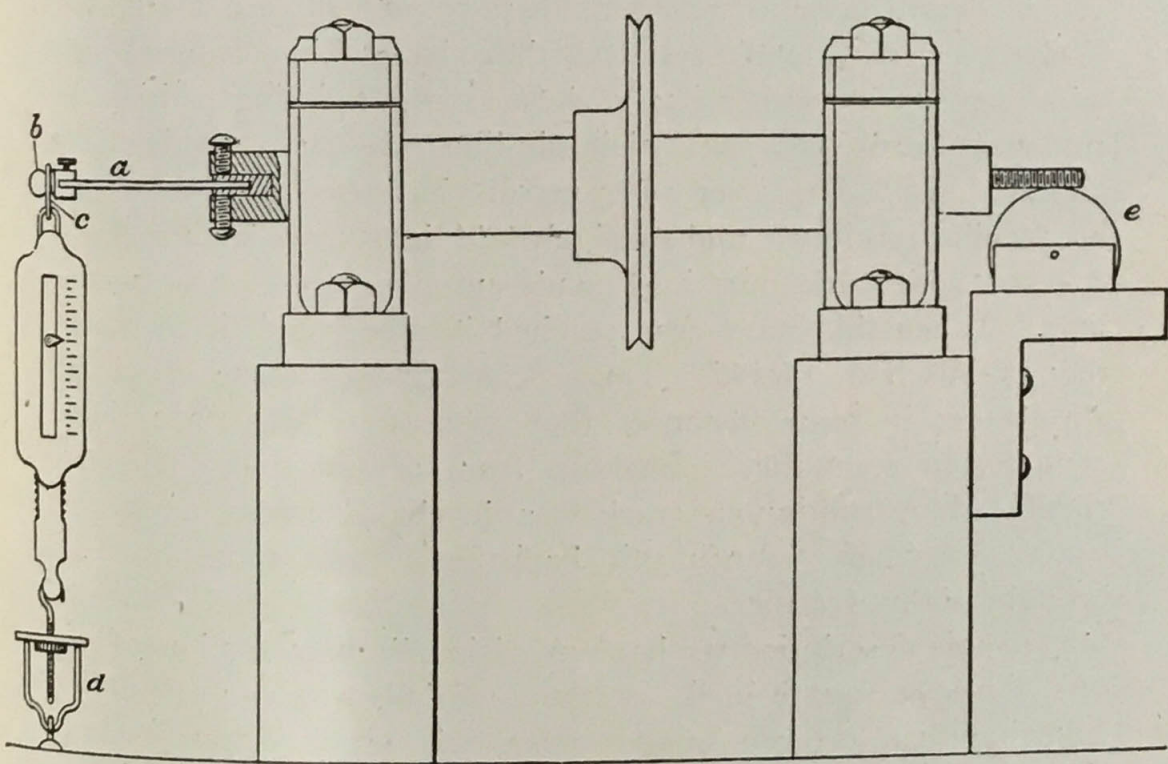


Fig. 2.

its neighbors. Nothing of the kind was seen, though the boundaries in some instances could be traced through the development in different directions of slip-bands over individual neighboring crystals. And later experiments, which will now be described, demonstrated that the mischief which is done by repeated straining occurs in quite a different way. In experiments made with stresses ranging from 14 down to 9 tons per square inch it was found that fracture ultimately resulted in all cases. The course of the breakdown was as follows: The first examination, made

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\* Ewing and Rosenhain, loc. cit., p. 372.



after a few reversals of the stress, showed slip-lines on some of the crystals, on many of them, if the stress was comparatively great, but on a few only if the stress did not much exceed the lower limit named above (of 9 tons per square inch). At this early stage the slip-lines were quite similar in appearance to those which are seen when a simple tensile stress exceeding the elastic limit is applied. Viewed under vertical illumination they appeared as fine dark lines. After more reversals of stress additional slip-lines appeared, which had not been visible in the first instance, but the most conspicuous feature was that those which were visible before became far more distinct and showed a tendency to broaden. After many reversals they changed into comparatively wide bands with rather hazily defined edges, losing entirely the fine and sharp character which slip-lines present when they first appear. As the number of reversals increased this process of broadening continued, and some parts of the surface became almost covered with dark markings made up of groups of broadened lines. When this stage was reached it was found that some of the crystals had *cracked*. The cracks occurred along broadened slip-bands; in some instances they were first seen on a single crystal, but soon they joined up from crystal to crystal, until finally a long continuous crack was developed across the surface of the specimen. When this happened a few more reversals brought about fracture.

In this description we have provisionally named a lower limit of 9 tons per square inch, but the experiments give grounds for believing that an even smaller stress will produce fracture in a similar manner if the process of reversing the stress is continued sufficiently long. There is clear evidence that with 9 tons per square inch fracture results. But we have also observed that with 8, and even 7, tons per square inch slip-lines appear, and that after many reversals they become accentuated and broaden in the manner that has been described. There is, therefore, a strong presumption that reversals of a stress of 8, or even 7, tons would ultimately develop cracks in the same manner as they are developed by stresses of 9 tons per square inch and over.

The destructive process, of which the above is a brief account, is illustrated in the accompanying micro-photographs (Plates 7-9).

The first series show part of a specimen which had been subjected to a stress the maximum value of which, close to the



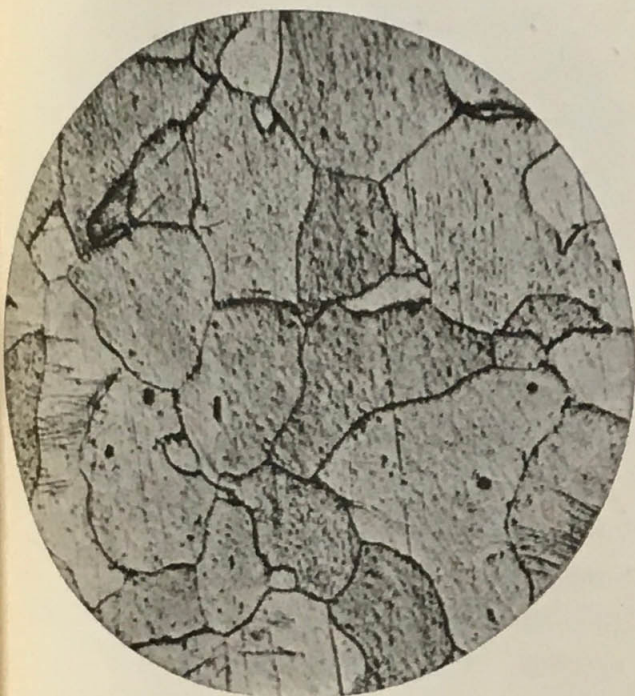


Fig. 3. Specimen of Swedish Iron after 5,000 reversals of a stress of 14.3 tons per sq. inch .  
Magnified 150 diameters.

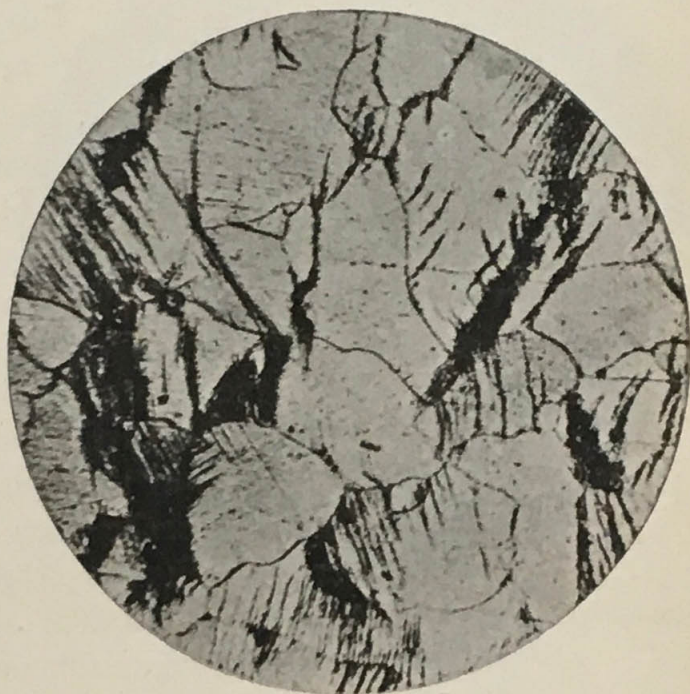


Fig. 4. Same after 40,000 reversals.  
Magnified 150 diameters.

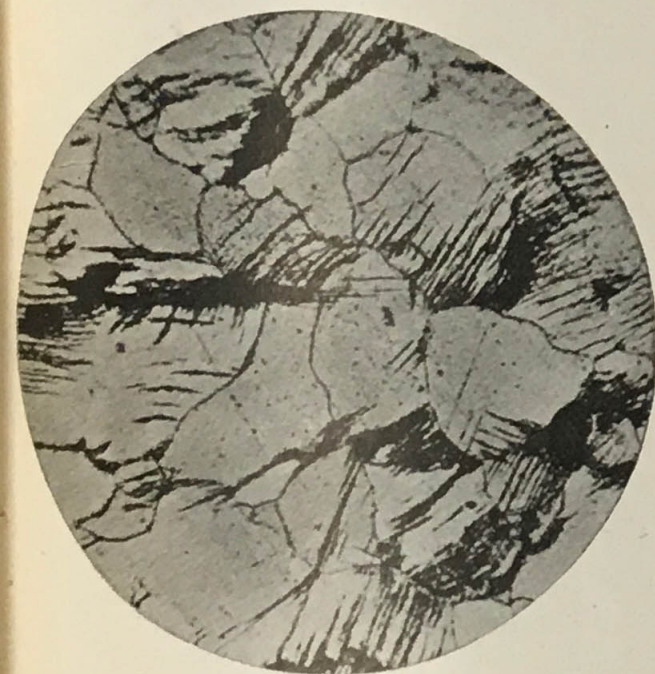


Fig. 5. Same after 60,000 reversals.  
Magnified 150 diameters.

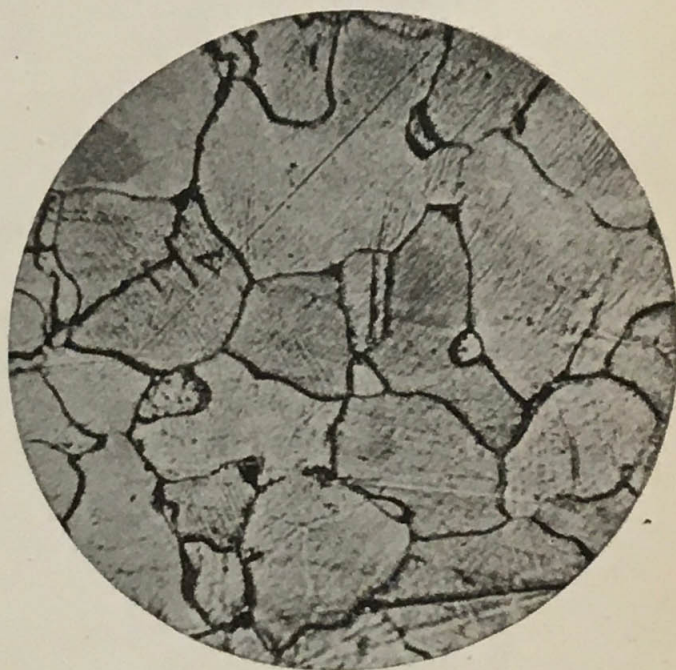


Fig. 6. Same after 70,000 reversals and re-polishing and re-etching.  
Magnified 150 diameters.



grips, was 14.3 tons per square inch. In this series the magnification is 150 diameters. Fig. 3 was taken after 5,000 reversals had been given. A few of the crystals exhibit signs of slip, the slip-lines being still fairly fine and sharp. Two crystals in the photograph are seen to have yielded more than the others. Figs. 4 and 5 show the same set of crystals after 40,000 and 60,000 reversals of stress respectively. In Fig. 4 a good many more crystals show signs of slip, and the slip-lines which appeared in Fig. 3 are far more strongly defined. In some cases it will be seen that the lines have so broadened out as to run together and form dark patches on the surface of the crystals. In Fig. 5 a still further breaking up of the surface by slip has occurred. At this stage, and probably earlier, some of the slip-bands have developed into small cracks. Such a crack is seen near the top right-hand corner of the figure, first of all close to a crystalline boundary, and then in steps across the next crystal. On the latter crystal two systems of slip-bands had formed at right angles to one another and at about  $45^\circ$  to the direction of stress. Only one system is clearly visible in the photograph, but it will be seen that the crack runs in steps along both. Fig. 5 shows not only the development of this crack, but also a general increase in width, length and number of the slip-bands. This was practically the final stage, so far as this portion of the surface was concerned, for the specimen broke after another 10,000 reversals along a crack outside the field of these photographs.

When viewed in this state it is not practicable to tell how many of the slip-bands have actually developed into cracks, but this is readily seen when the specimen is re-polished and reëtched. This treatment obliterates any ordinary slip-bands which are steps marking differences of surface level but any cracks remain visible. The specimen which gave this series of photographs was accordingly re-polished sufficiently to clear away the slip-bands, and was reëtched. This left the cracks alone visible, rather accentuated indeed, for the sides of the cracks are to some extent eaten away by the acid, and hence the width of the crack is increased. Fig. 6 shows the same part of the specimen as Figs. 3, 4 and 5 after re-polishing and reëtching. The slip-bands have disappeared, except where they have formed cracks. A careful comparison of this with Fig. 3 will show where cracks have formed. The most conspicuous crack is at *AA*, and its zigzag character as it

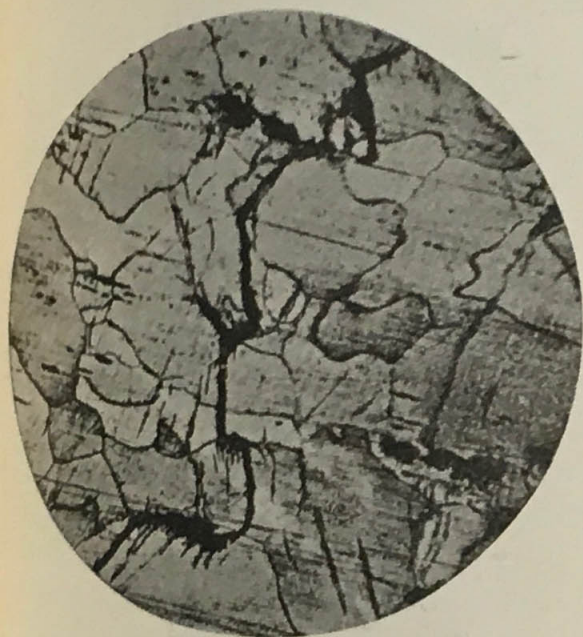


Fig. 7. Specimen after 170,000 reversals of a stress of 12.3 tons per sq. inch.  
Magnified 150 diameters.

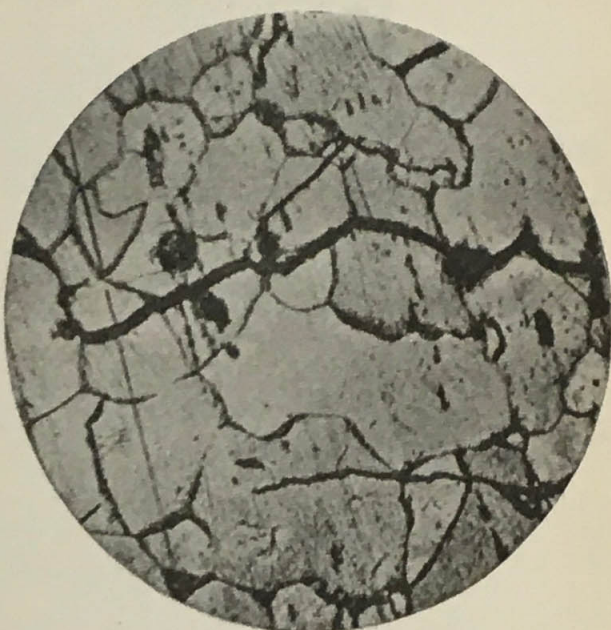


Fig. 8. Same after re-polishing and re-etching.  
Magnified 150 diameters.

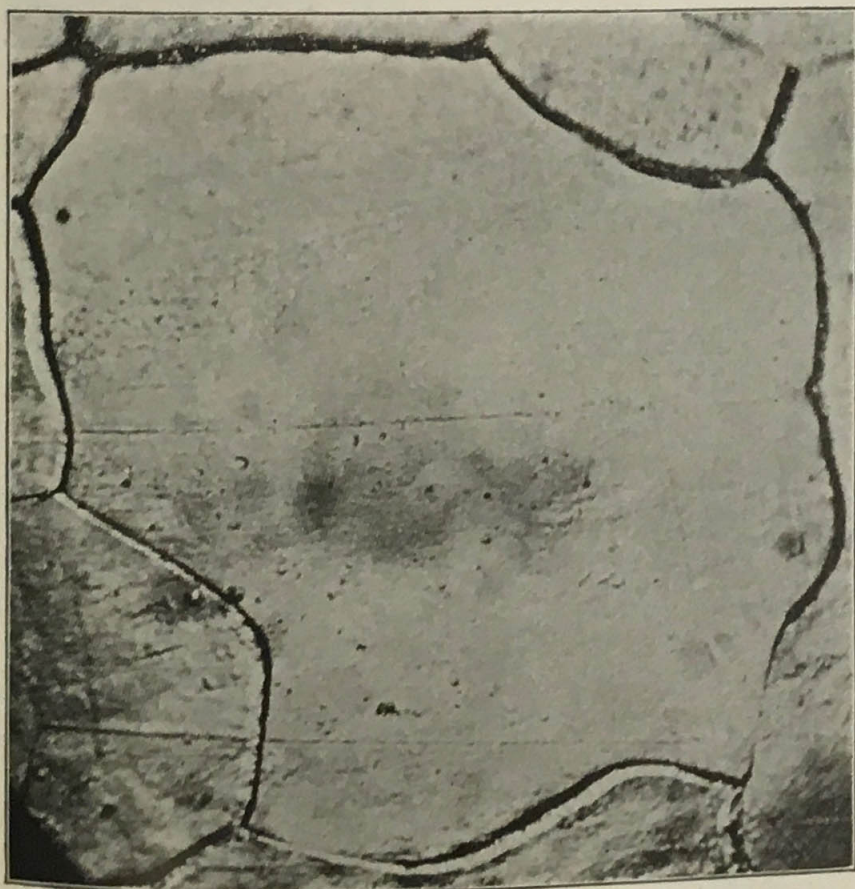


Fig. 9. Specimen after 1000 reversals of a stress of 12.4 tons per sq. inch.  
Magnified 1000 diameters.





Fig. 10. Same after 2000 reversals. Magnified 1000 diameters.

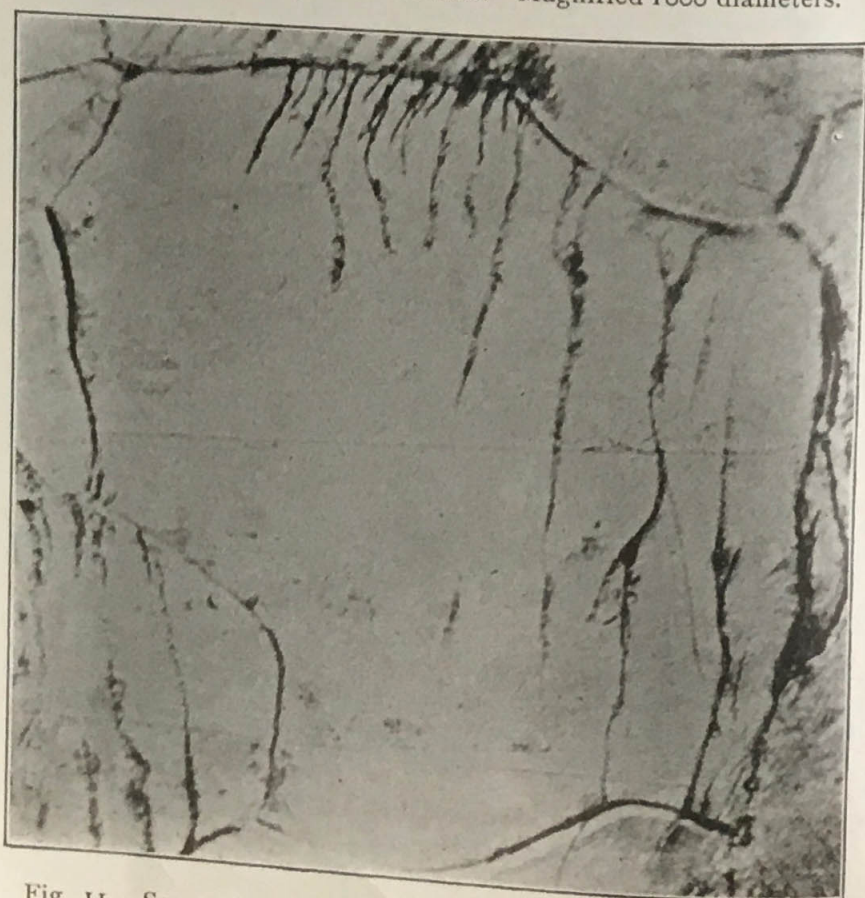


Fig. 11. Same after 10,000 reversals. Magnified 1000 diameters.

follows the two directions of slip-bands across one of the crystals is specially noticeable.

The specimen illustrated in the next photograph, Fig. 7, had been subjected to 170,000 reversals of a stress of 12.3 tons per square inch, at which it broke. The spot shown (magnified 150 diameters) is a little way further from the grips than the crack through which the specimen actually broke, but another severe crack is seen running across the centre of the figure. Comparing this with Fig. 5, it will be at once seen that there are far fewer lines due to slip upon each individual crystal than in the former specimen. Fig. 8 shows the same spot after re-polishing and etching. Comparing this with Fig. 6 it is seen that a greater proportion of the slip-lines appear in this instance as cracks after re-polishing. The maximum stress is less here than in the former example, and more than twice as many reversals were required to bring about fracture. This agrees with what has been found for all other specimens broken, viz., the lower the stress the fewer the slip-lines upon each crystal, but the greater proportion of these actually develop into cracks under the more numerous reversals to which the less severely stressed specimen is exposed.

The photographs described above are on rather too small a scale for the actual changes in the slip-lines themselves to be clearly seen, and these are better illustrated by the next series, Figs. 9-12. These show with a magnification of 1000 diameters a small part of the surface of another specimen of the same iron which was subjected to reversals of 12.4 tons per sq. inch. Fig. 9 is taken after 1000 reversals. The slip-bands which have formed are very faintly visible as fine lines upon the surface of the crystal. Fig. 10 is after 2000 reversals; the slip-bands seen in Fig. 9 are now more distinct, and some new ones on the right of the crystal are fairly strongly defined. Fig. 11 is after 10,000 reversals; some of the slip-bands now show a decided tendency to broaden out and those upon the right have extended further across the crystal. Fig. 12, taken after 40,000 reversals of stress, shows further broadening out and spreading of the slip-bands. At this stage it could be seen by the focussing that this broadening was due to a heaving-up of the surface of the crystal in the neighborhood of each slip-band, the markings being decidedly above the level of the other parts of the crystal. It is to be conjectured that the action is of the kind indicated in the





Fig. 12. Same after 40,000 reversals. Magnified 1000 diameters.

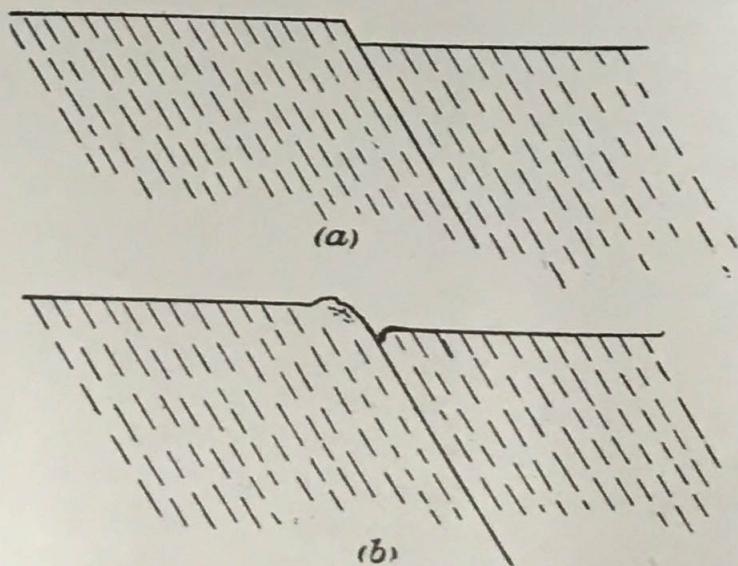


Fig. 13.

sketch (Fig. 13) where *a* represents an ordinary slip-band seen in section at right angles to its length, and *b* represents the effect of reversals of stress upon it. Very little further change took place in the particular crystal of Figs. 9-12 as further reversals were applied, the specimen breaking elsewhere after 160,000 reversals. It has been noticed in this respect that when once an incipient crack begins to form across a certain set of crystals, the effect of further reversals is mainly confined to the neighborhood of the crack, other crystals (as was the case with that illustrated in Figs. 9-12) changing but slightly.

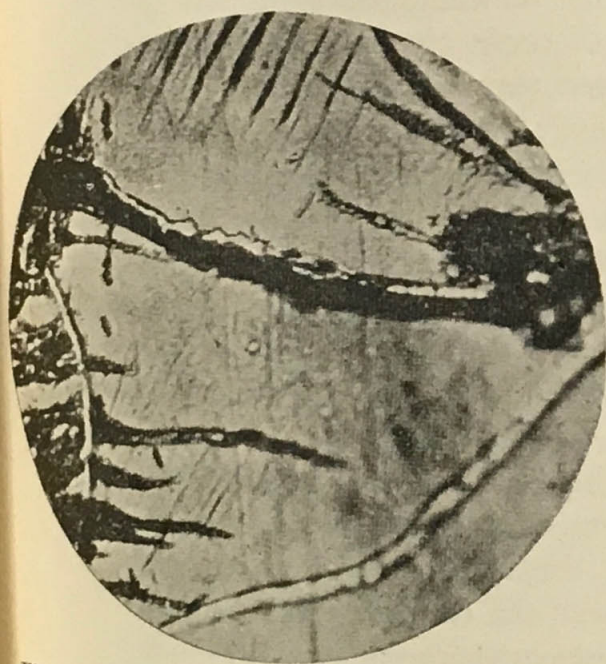


Fig. 14. Another part of the specimen of Figs. 9-12, after 160,000 reversals. Magnified 1000 diameters.

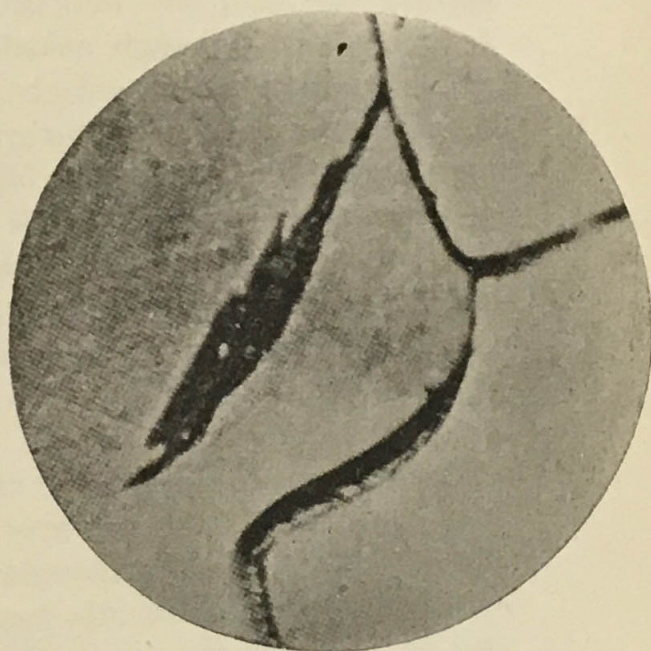


Fig. 15. Specimen after 3,000,000 reversals of a stress of 6.9 tons per sq. inch. Magnified 1000 diameters.

Fig. 14 (Plate 8) shows a different part of the same specimen (after it had broken), also under a magnification of 1000 diameters. The broad band in the middle of the crystal is a crack which has developed along what was originally a line of slip. The heaving-up of the surface along the edges of the crack is well marked and may be compared with similar appearances at the edges of slip-bands in other parts of the photograph.

The stresses which are stated here are in all cases calculated from the observed load, as measured by the spring balance, acting at the end of projecting beam or "cantilever," and they are the values which (on the ordinary theory of bending) are reached



at a place close to the clamp. It was observed, however, that the destructive effects of reversals were not confined to the metal immediately adjacent to the clamps, but extended in most cases for a considerable way towards the loaded end of the specimen. The development of slip-bands, and their gradual widening and final conversion into cracks, occurred in some cases at least half an inch from the clamp, at a place where the fixing of the specimen could not disturb the distribution of stress in any way.

In another experiment the load was such as to produce a maximum stress, close to the clamp, of 9.2 tons per sq. inch, and 800,000 reversals were given. It was then seen that the greater number (though not all) of those crystals which closely adjoined the place where the specimen was clamped showed signs of repeated slip. Further away from the clamp the slip-lines became less numerous; but they were plainly seen on individual crystals as much as half an inch from the clamp. At the most distant places where slip-lines were plainly apparent the stress was only 7.3 tons per sq. inch. It was clear that a stress no greater than this was sufficient to develop slips, under many reversals, and that the lines so produced became accentuated as the process went on.

This was confirmed by another experiment in which the maximum stress, close to the clamp, was only 6.9 tons per sq. inch. After 3,000,000 reversals of this stress *one* slip-band was observed on a crystal a little way from the clamp. This is shown in Fig. 15, where the slip-band is seen in the broadened condition which resulted from 3,000,000 reversals of stress. Prior to this, the same specimen had suffered 1,000,000 reversals of a stress of 5.3 tons per sq. inch, without showing the smallest sign of damage. It was only after increasing the stress to 6.9 tons per sq. inch that any action became apparent. It is an open question whether an isolated slip-band such as this would have led to fracture, if the process of reversal had been continued. At the conclusion of the experiment it was still confined to one crystal and it did not even extend all the way across that.

We have noticed that when lines indicating slip appear during reversals of a comparatively small stress, they are generally to be found in the central parts of individual crystals, not extending to the boundaries of the crystal.

It appears, then, that this material suffers no damage from



repeated reversals of a stress of 5 tons per sq. inch; but that when the stress is raised to 7 tons per sq. inch signs of fatigue are apparent after many reversals. And further, that with a stress of no more than 9 tons per sq. inch, the damage caused by reversals is so considerable that cracks are formed and the piece breaks. In all probability fracture through the formation of cracks would occur with 7 tons also, though all that is actually demonstrated for this stress is that it causes slip-bands to appear and to become accentuated in the manner which, with greater stresses, leads to the development of cracks.

It is remarkable that these actions are brought about by stresses much below what is ordinarily understood by the elastic limit of the material. A tensile test shows proportionality of strain to stress up to 12 or 13 tons per sq. inch, with no apparent defect of elasticity. The conditions under which these experiments were made seem to exclude the possibility that vibration gave rise to local augmentation of the stresses. The manner in which the slip-lines appear shows that some crystals reach a limit of elasticity sooner than others. This is no doubt to be ascribed in part to their being so oriented that the gliding planes, on which slip occurs, are inclined in the direction which is most favorable to the action of tangential stress. But besides this, it may be conjectured that in a complex structure built up of many crystals irregular in form the distribution of stress from crystal to crystal is by no means homogeneous.

Whatever the selective action of the stress is due to, the experiments demonstrate that in repeated reversals of stress certain crystals are attacked and yield by slipping, as in other cases of non-elastic strain. As the reversals proceed, the surfaces on which slipping has occurred continue to be surfaces of weakness. The parts of the crystal lying on the two sides of each such surface continue to slide back and forth over one another. The effect of this repeated sliding or grinding is seen at the polished surface of the specimen by the production of a *burr*, or rough and jagged irregular edge, broadening the slip-band, and suggesting the accumulation of *débris*. Within the crystal this repeated grinding tends to destroy the cohesion of the metal across the surface of the slip, and in certain cases this develops into a crack. Once the crack is formed it quickly grows in a well known manner, by tearing at its edge, in con-



sequence of the concentration of stress which results from lack of continuity. Engineers are familiar with the development of cracks, even in the most ductile materials, when these are initiated at air bubbles or other flaws. The present experiments show how a crack may be formed, without any flaw to serve as nucleus, the first breach of continuity being set up through repeated grinding on a plane of slip in perfectly sound metal.

The experiments throw light on the known fact that fracture by repeated reversals or alternations of stress resembles fracture resulting from a "creeping" flaw in its abruptness and in the absence of local drawing-out or other deformation of shape. They also help to explain why it is that a piece that has been subjected to many reversals shows no apparent loss of strength or plasticity when subjected to an ordinary tensile test. So long as the reversals have not yet reached the stage of producing cracks, it is not to be expected that such a test will detect the deterioration which has occurred. The material will still yield by slipping much as at first, and neither its plasticity nor its strength need show much change.

Interesting points suggest themselves which require further investigation. It is well known that when a plastic metal such as iron is strained sufficiently to take permanent "set," it suffers a temporary loss of elasticity, which is recoverable by lapse of time, the recovery of which, as Muir has shown, may be enormously accelerated by warming the piece to such a temperature as  $100^{\circ}$  C. This may be ascribed to a gradual healing action which restores the resistance to sliding on the planes of slip after they have been weakened by the first severe strain. The first strain makes subsequent slipping easier, for a time, but when the material has a long enough rest recovery ensues. Probably enough, a similar recovery would occur if during the application of reversals of stress long intervals of rest were allowed, and still more if during these intervals the temperature of the piece were raised. It may be conjectured that such treatment would arrest the destructive process of fatigue in its earlier stages, and give the material a new lease of life. The damage which alternating stresses produce probably depends not only on the amount of the stress and the number of alternations it suffers, but on the rapidity with which the alternations follow one another, and on the continuity or otherwise of the alternating action.



## THE ROLLING AND STRUCTURE OF STEEL RAILS\*

By P. H. DUDLEY

**I**N compliance with your request to supplement my report upon recent practice in rolling rails, with additional data, I am revising and elaborating portions of my first report. This is for a more extensive study of the structure of the steel rails now in service, and also the structure of the high-grade metal in the splice bars, which has rendered such efficient service the past few years.

Several features in the manufacture of the rails which were only mentioned in the report, will be more fully considered.

The structure shown in the photo-micrographs will be computed, and stated as so many thousand granulations per square inch. In this way it will be easier to compare the structure of the sections which wear well under the wheel treads, as well as the structure which is essential for girders, with high elastic limits. This will group the structure of the different steels, so the relations to wear, high elastic limits, and hardness with toughness, should be apparent, provided they exist as generally expected.

The following is a brief report of my visit to the steel mills last summer, to make some rails for you, and to study their latest practice in the manufacture and colder rolling of rails.

The coal strike was in effect, and only a few thousand tons of rails of the orders were obtained from the last of May to the first of September.

The physical properties of the rails incident to the colder rolling, were modified. The elastic limits of the steel were lowered, so as to affect the section for high-speed service.

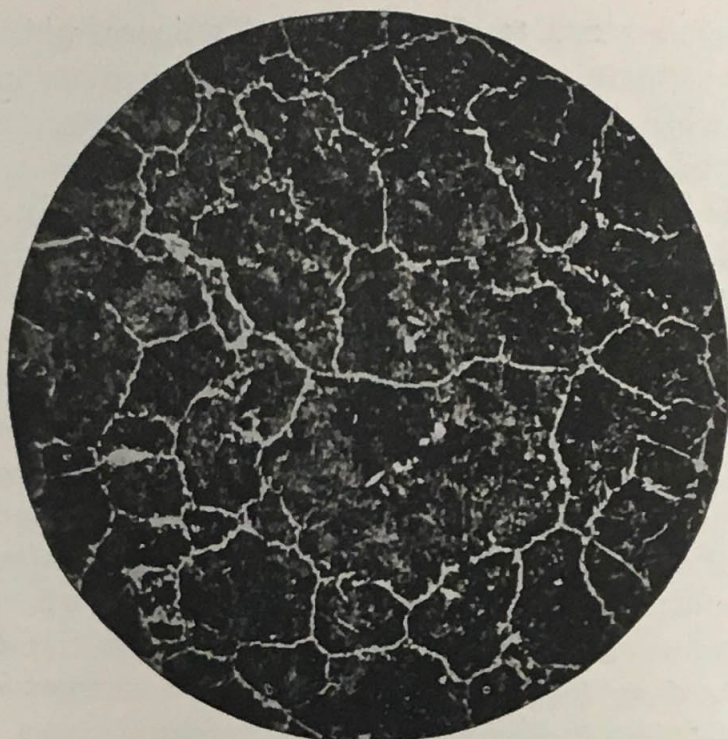
Several thousand tons of rails were rolled in December, in which I made a slight change in composition, to improve the physical properties under colder rolling. This gave a fine granular structure and nearly the same physical properties as formerly obtained in rails which stand up in the track, under present service, as girders.

I visited first the Pennsylvania Steel Company's mill at

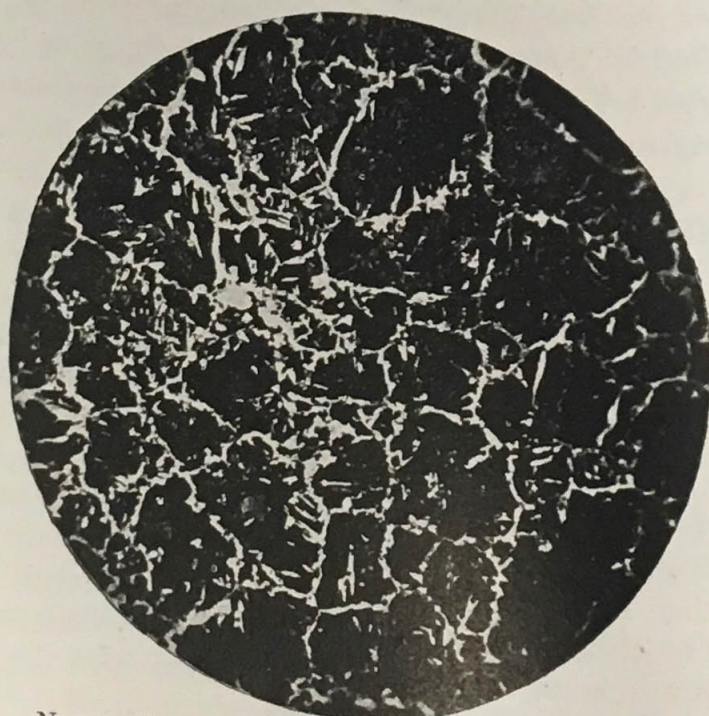
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\* A report to W. J. Wilgus, esq., 5th Vice President, New York Central and Hudson River Railroad Co., dated January 19, 1903.





No. 1. Coarse Granular. 85 lb. Sec. P. S. Co., 1902.  
Magnified 50 diameters. 2,000 Granulations per sq. inch.  
17 x 20 inches Ingot on Base.



No. 2. Coarse Granular. 85 lb. Sec. P. S. Co. 1902.  
Magnified 50 diameters. 2,500 Granulations per sq. inch.  
17 x 20 inches Ingot on Base.

both 85-lb. rails, the number of granulations per square inch being low in this series.

At Steelton the Pennsylvania Company would like to roll only rails of 70 lbs. or less; the heavier sections going to Sparrows Point.

At the Maryland Steel Company's mill of the Penna. Steel Company, at Sparrows Point, the practice in some respects is quite different. Direct metal is used for the converters, from the blast furnaces, practically the same as at Steelton, when they are running under normal conditions. When I was there making your rails, there was not sufficient fuel or iron to keep the mill running on Saturdays, so that the metal produced by the furnaces on Saturday and Sunday was taken in vessels to a series of moving casting pans, and as the fluid iron charges the pans, with about 100 lbs. or more of metal, they are submerged immediately in water, and in less than a hundred feet of travel the metal becomes solid and is dumped into cars. Casting the metal into pigs, in sand, as formerly, is obviated. The iron is clean, and in this respect is decidedly advantageous.

The cast metal is remelted in cupolas, as was formerly the practice of all steel mills until a recent date. The steel converters are eighteen tons' capacity, and with the silicon content controlled in the iron, but little scrap is used, the blowing being finished in nine to eleven minutes. The bath is blown full, as stated in our specifications, though not "over-blown." The silicon content for the steel, however, is less than there specified. Direct metal with the silicon content controlled for the converters, less scrap charged, renders the blowing more uniform, and is an advance on the practice of only a few years since.

The bath is recarburized, as already described at Steelton, and then an electric overhead crane, carrying the teeming ladle, receives the charge of metal, and the ingots teemed in moulds on cars.

Mr. F. W. Wood, president of the Maryland Steel Co. at Sparrows Point, originated the practice of dispensing with the pit-casting, and teeming the metal for ingots in moulds on cars, in 1890. Also the mechanical stripper. Both features are so desirable practice, that they have become general in the large plants in the United States, the ingots being better from the metallurgic standpoint.



The present ingots are  $20 \times 21$  inches on the base, and with a taper of little more than one inch in six feet; and for one or two minutes after the mould was charged, there was a gentle ebullition on the top from the sides of the mould, then the metal set quiet, and was capped.

The moderate drafts of the moulds at Sparrows Point, as at Steelton, is a great advantage in facilitating the escape of gases and impurities from the junctions of the columnar structure in the corners of the moulds. In this respect they are more favorable than moulds of greater draft for hand-stripping. This will be of decided benefit, checking irregular detrusion of metal from the top corners of the rail-heads, under the intensity of pressure of the present wheel-loads.

The ingots on the cars, in about 15 minutes, go to the hydraulic stripper, are stripped, and then charged into vertical heating furnaces.

The ingots are retained in a vertical position, for all manipulations of stripping and heating, until ready for blooming. The air and gas in the heating furnaces are so controlled that the "heaters" cannot rush the heating. The time required for equalizing the heat is an hour and a quarter to an hour and twenty minutes. It is automatic, under the control of the mill superintendent, and the "heaters" are checked frequently by photomicrographs of the steel, to prevent over-heating, causing a coarse granular structure.

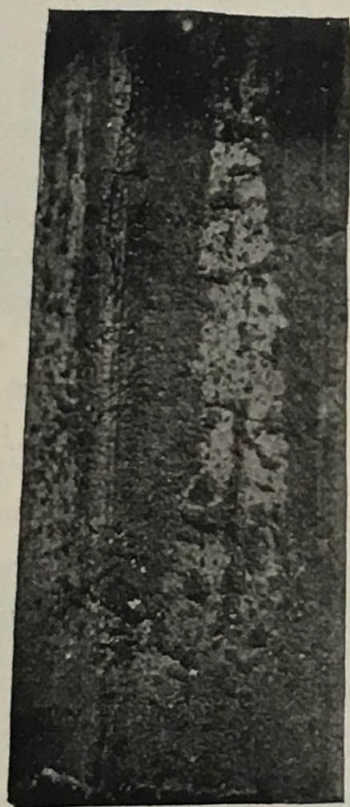
This is really heat treatment commencing with the ingot.

The ingots, as taken from the heating furnaces, do not show dripping cinder, are evenly heated, and bloom straight, and not curved, owing to one side being hotter than the opposite. An even scale is shown over the entire surface, as you will notice from the accompanying photograph, marked 3, of a red-hot ingot, reduced 25 times. This heat treatment of the ingots cut the "seconds" from 8 to under 5 per cent, your rails running under 4 per cent.

From the heating furnace the ingots go to the blooming mill, and heat treatment is continued. After the structure of the skin of the ingot has been consolidated in two or three passes, then, on the portion of the bloom which is to form the head of the rail, a stream of water is forced, cooling this more rapidly than the other portions of the ingot. For an instant this portion becomes

darkened, but is reheated immediately by the escaping heat of the ingot, which dissipates the heat units of the steel in a more rapid manner than is possible by the ordinary blooming train. This feature of cooling the bloom is subject to a patent.

From the blooming train, the bloom goes to the shears, is cropped and cut into lengths for rolling two or three lengths of rails, according to the weight of the section. The blooms are not re-heated, but one piece goes directly to the roughing train, and



No. 3. Photograph of Red Hot Ingot.  
20 x 21 inches on Base. M. S. Co., 1902. Reduced 25 times.

then to the intermediate train. The second piece is delayed a few seconds, then follows the first piece. This method prolongs the time of rolling each rail for colder working of the steel, without restricting the output.

The finishing train has but one pass, and is distinct by itself.

In all these operations, the metal is being delayed, the structure refined and cooled as it passes through the various trains to the finishing train, at a low temperature, for the short shrinkages now prescribed.



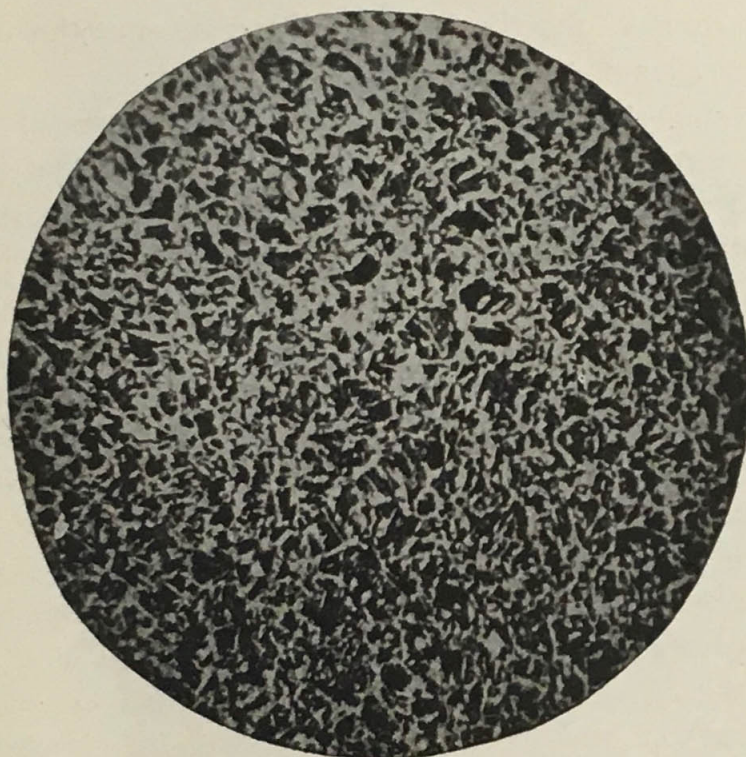
There are several features of colder rolling of the rails, and shrinkages, which require considerable attention. While a gain is made in refined structure, it may be at the expense of other physical properties quite as important. In the operations at Sparrows Point, the metal for the rail section has been cooling from the time that it entered the blooming train until it passes through the finishing train, the metal being well worked and the structure fine. The shrinkage limit itself prescribed for the last pass to govern the structure will not control it; while it may control the proper shrinkage, the structure may be so large in the central portion of the rail that it is not broken or refined in the last pass of the train. The structure of the metal must be checked and restricted in forming, long before the last pass, to make this fine and uniform throughout the entire section of the rail.

Another feature of this method of heat treatment is that, with our sections as designed now, the head is cooled so much faster, while the other portions contain a sufficient amount of heat, so that the rolling temperatures are sufficiently uniform throughout the entire section. For colder rolling, it will not be necessary to change the form of section, when a proper heat treatment is observed during the manufacture of the rails.

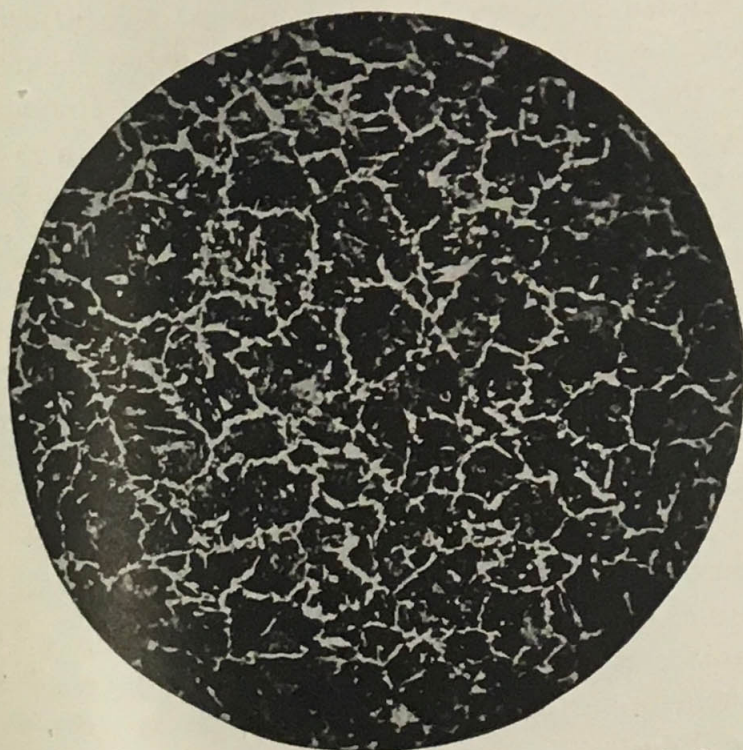
From the hot saws, the rails travel a long distance to the hot bed, which spaces each rail 6 inches apart, for the ordinary 5-inch section; it would be slightly less for a section 6 inches in height. The rails are cooled uniformly, all curved nearly alike. In making your rails, I had them slightly under-cambered, so that, when cool, in 33 feet they had a parabolic curve of about  $\frac{1}{4}$  to  $\frac{1}{2}$  inch versed sine. The pressure to straighten the rails on the surface was not severe, nor that upon the side. Four to six light blows on the base were all that was required to straighten the rails smooth, with perhaps a like number upon the sides. As to the effectiveness of the method, you have noticed that by the rails which have been laid in the track. This method has been adopted now for all sections.

New forms of hardened steel "fullers," with wooden handles, have been designed and put into service by Mr. S. S. Martin, superintendent of steel department of the Maryland Steel Company. The faces of the fullers are broad, and have rounded corners, so that indentation of the surface of the head of the rails is obviated practically. This is essential to produce rails having





No. 4. Non-Granular. 80 lb. Sec., N. Y. C. & H. R. R. R., M. S. Co., Sept., 1902.  
Magnified 50 diameters.  
Incomplete Granulations, 10,000 per inch. Ingot on Base.



No. 5. Granular. 80 lb. Sec., N. Y. C. & H. R. R. R., M. S. Co., Sept., 1902.  
Magnified 50 diameters.  
4,500 Granulations per sq. inch. 20 x 21 inch Ingot on Base.



a smooth surface, for the high standard of smoothness required now for fast trains.

In testing the butts for the physical properties of these rails which were rolled so cold, it was noticed at once that the deflection sets were increased decidedly over those which were rolled at a slightly higher temperature, or the structure had been fixed by composition.

The standard deflection set for the  $5\frac{1}{8}$  inch 80 lb. rails, moment of inertia 28.5 4th power inches, is 1.55 inches for 2000 lbs. falling 20 ft., supports 3 ft. centers. This indicates the essential physical properties requisite to stand up in the track as girders, for your service.

The colder rolled rails had deflection sets of 2.25 to 2.5 inches, which was too great reduction in the elastic limits.

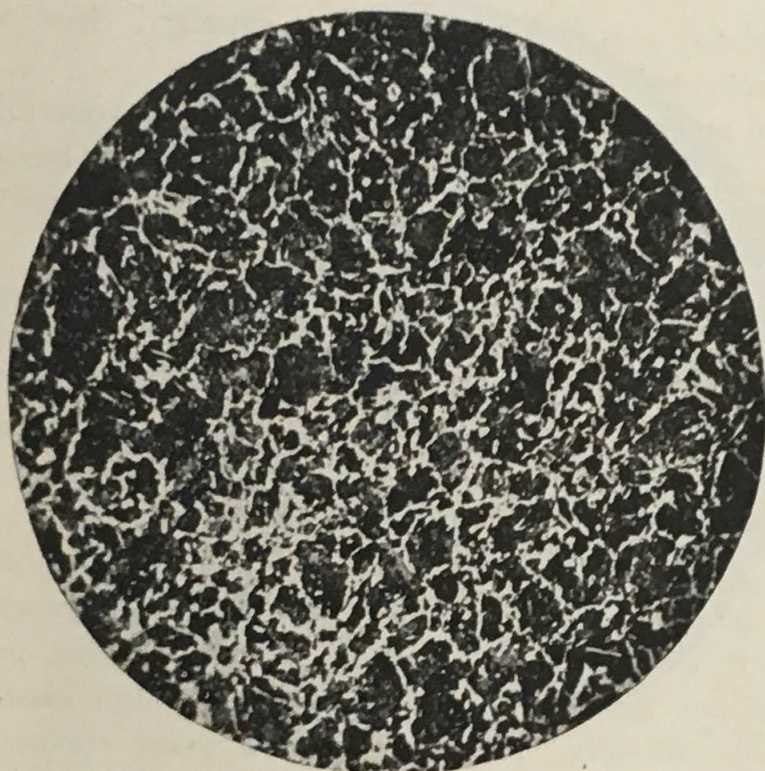
One advantage of the nickel steel rails is that with a refined structure the elastic limits are even higher than are obtained in the high carbon rails. A hardness is obtained, combined with great toughness and high elastic limits, which are the characteristics, though in a lesser degree, of the rails which have rendered such excellent service in your tracks.

The refined structure of the metal at once affects the rail as a girder, and it is not desirable to lower the elastic limits so as to have them exceeded by the rapid moving engines exerting their present tractive power as they pass over them in the track. The unit fiber strains in the base of the  $5\frac{1}{8}$  inch 80 lb. rails under the highest speed trains often reach 40,000 to 45,000 lbs. even when the track is in what may be termed the best condition. In the rails rolled in December, I secured high physical properties, with fine structure. While the latter is to be desired, we must conserve the physical properties essential for a girder, as well as to sustain the wear of the head of the rail. The essential conditions for each are not incompatible in the same section.

To indicate the structure attained in the rolling of the rails, the photo-micrograph marked "Non-Granular, No. 4," from my "Questions for the International Railway Congress," shows an excellent structure, though the elastic limits are not as high as I have found desirable for high-speed trains on 80 lb. rails.

The one marked "Granular, No. 5," is one of the coarsest found in the rails rolled in September.

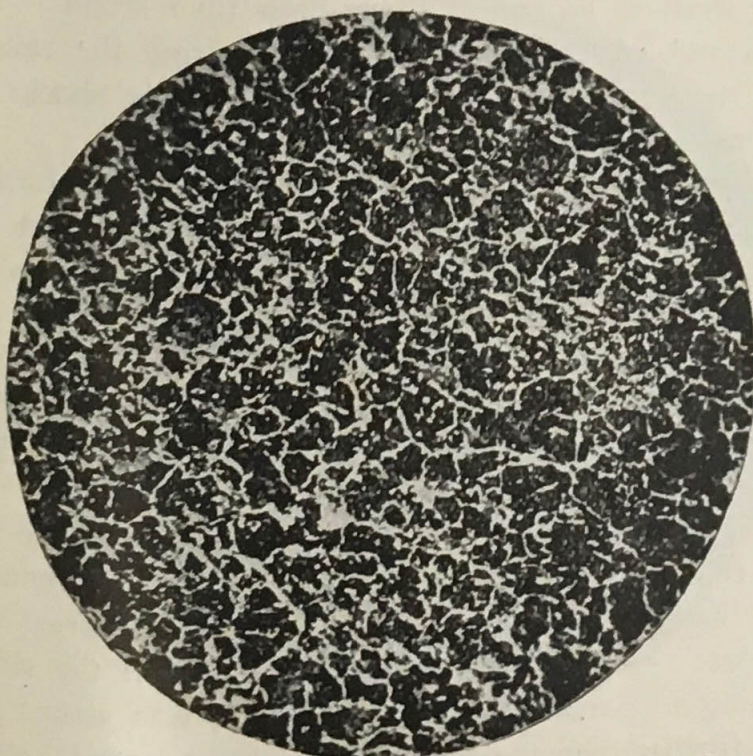




No. 6. 80 lb. Scc., N. Y. C. & H. R. R. R. M. S. Co., November, 1902.

Magnified 50 diameters.

6,000 Granulations per sq. inch. 20 x 21 inch Ingot on Base.



No. 7. 80 lb. N. Y. C. & H. R. R. R., M. S. Co., November, 1902.

Magnified 50 diameters.

7,000 Granulations per sq. inch. 20 x 21 inch Ingot on Base.



As already stated, the "Coarse Granular," Nos. 1 and 2, are from reheated blooms.

Nos. 6 and 7 show the average structure of your rails rolled in August. The service tests of these rails in the track will be watched with great interest. From the excellent finish of the rails, the surface should wear smooth in the track.

From the character of the ingots produced, irregular detrusion of metal on the top corners of the rail heads should be limited to a few rails; only a slight general flow of the metal must be expected under severe service. From the structure of the metal, the rate of abrasion on inside of head of rails on curves should be slow.

In a subsequent rolling, photo-micrograph No. 8 shows the structure of a rail .54 in carbon and 1.20 in manganese. This structure is what I like for a girder, and for that percentage of carbon and manganese is excellent, has high elastic limits, considerable ductility, and will make a good surface-wearing rail for the present wheel loads, as well as a girder. A piece of this rail to be tested has been sent to the U. S. Testing Machine at Watertown, to find exactly its physical properties, also to make a rotating shaft to be tested under high fiber stress.

Frogs and switches are being made from the rails, to see how well the structure will stand the dynamic shocks incident to such service.

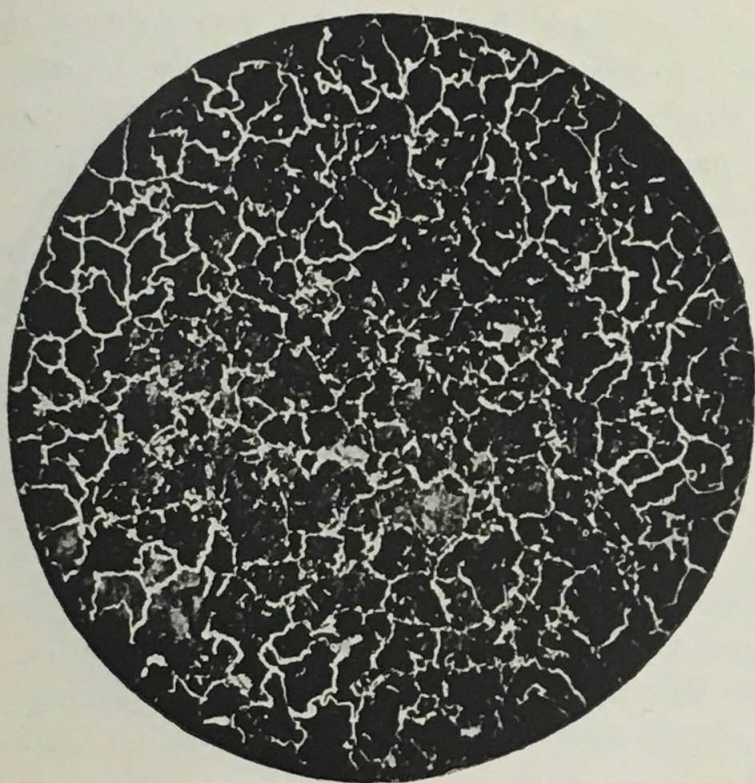
Photo-micrograph No. 9 is from one of the Lackawanna Iron & Steel Company's 65 lb. rails, direct rolling, made in 1890. This was in a frog in the yard at the Grand Central Station for over ten years, and not completely worn out.

Such service tests are of great value, and while the structure is not as uniform as is considered preferable now, the service rendered was good. The steel had elastic limits of 55,000 to 60,000 lbs. and was a good wearing rail.

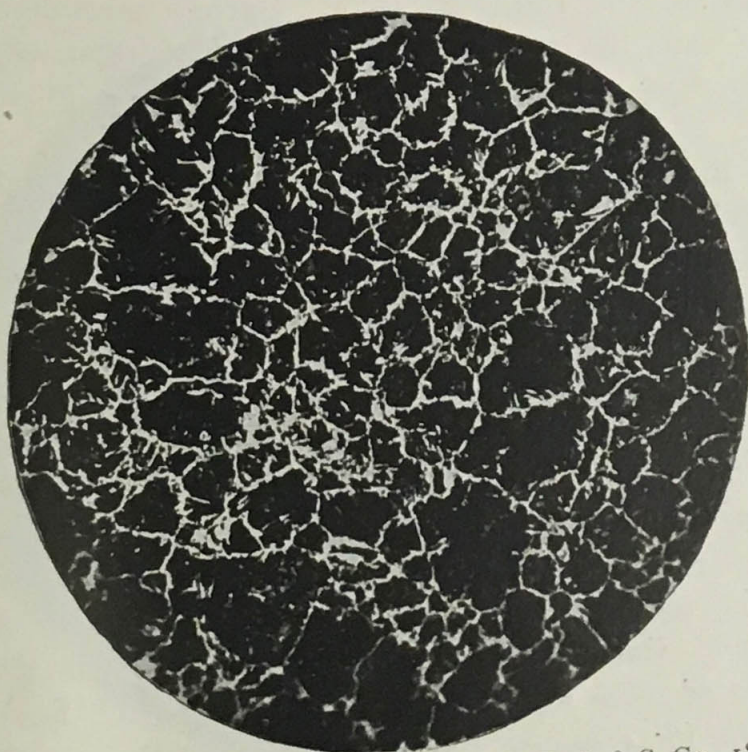
Photo-micrographs Nos. 10 and 11 are from the first 100 lb. rails rolled in 1892, and laid in the Park Avenue tunnel. This rail had been in service ten years, and carried over 150 million tons. A small piece was broken from the side of the head, owing to porosity of the ingot, and was etched. to see if anything further than the blow-hole could be traced.

Photo-micrograph No. 11 shows a streak of slag, extending through the center of the field of view. I did not make the





No. 8. 80 lb. Sec., N. Y. C. & H. R. R. R., M. S. Co., Nov. 1902. .54 Carbon and 1.20 Manganese. Magnified 50 Diameters. 6,000 Granulations per sq. inch. 20 x 21 inch Ingot on Base.



No. 9. 65 lb. Sec., N. Y. C. & H. R. R. R., L. I. & S. Co., 1890. Magnified 50 diameters. 5,000 Granulations per sq. inch. 14 inch sq. Ingot on Base.



first 100 lb. rails, and little attention was paid to the ingots. These were the first 100 lb. rails rolled in the United States.

The structure in photo-micrographs Nos. 9, 10 and 11 should be more uniform, like the fine granular of No. 8, for the best wearing rail and girder.

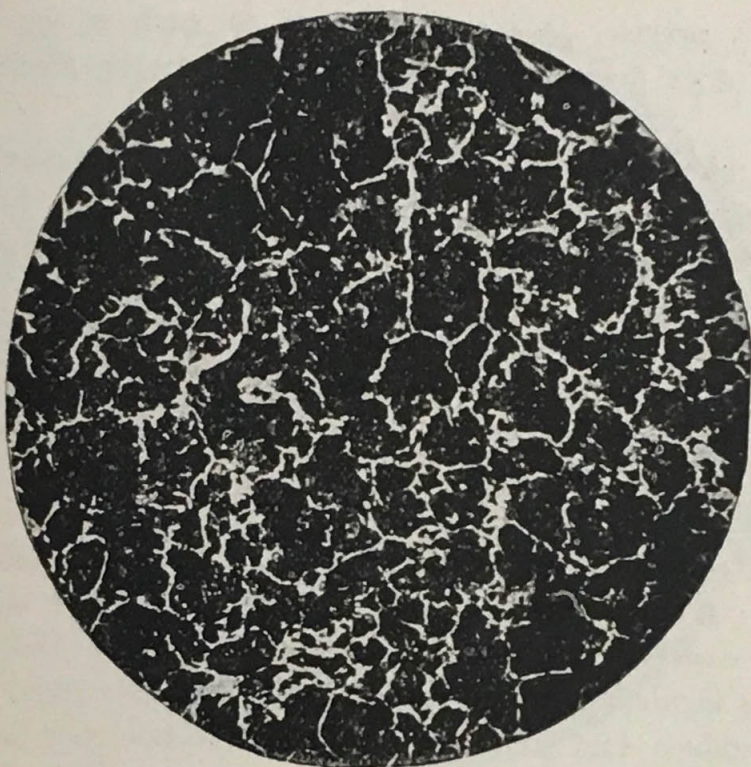
Photo-micrograph No. 12 shows the structure of one of the English Wilson Cammel rails, rolled in 1870. These rails did not do equal service in the track as those of the Charles Cammel, the Barrow, and the John Brown, which were purchased at the same time. The Works are not in existence now, and little information can be obtained in regard to the manufacture of the rails, but the structure does not indicate that the ingots were hammered. The rails were rolled evidently from very hot reheated blooms. For the low content of carbon and manganese, the structure should be classed Coarse Granular. The structure of the steel tells the story that the practice of making Bessemer steel rails, in the early date of its history, was not uniform, even in England.

Photo-micrograph No. 13 is from Barrow steel, 1873. This was a  $4\frac{1}{2}$  inch 65 lb. rail of the first N. Y. C. & H. R. R. R. 65 lb. section, and rolled in 1873. Barrow steel in the same section of rails has been found, of dates of 1870, 1871, 1872, 1873 and 1874. The structure will be shown in subsequent reports.

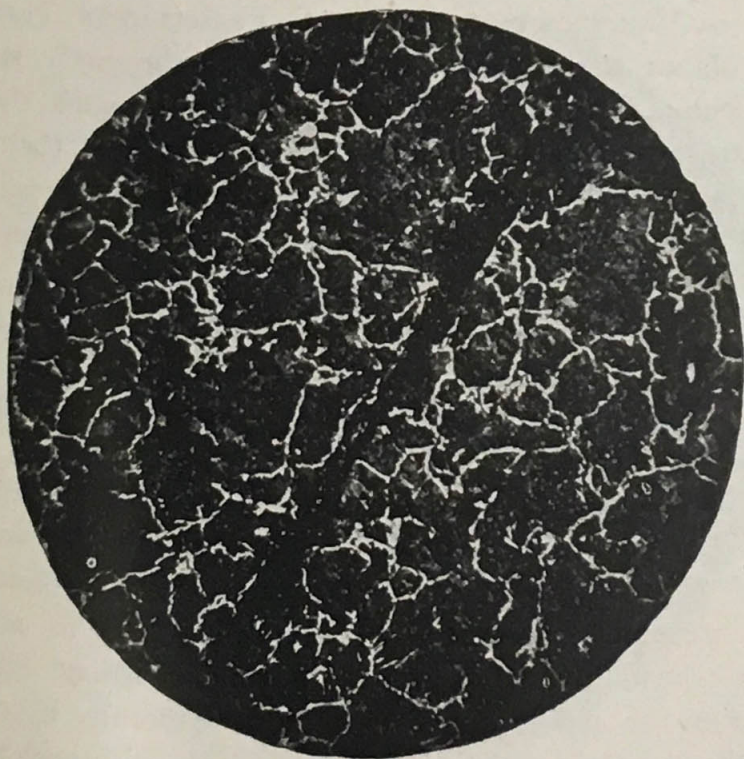
The Barrow steel rails from hematite ores, at that time, made excellent wearing rails in the track, but broke more frequently than the Cammel or the John Brown. They were made from small hammered ingots, and by the structure it is seen they were rolled at a low temperature, near or below the "critical range."

Photo-micrograph No. 14 is from a John Griswold rail, made at Troy, probably in 1874. This was from a small hammered ingot, and rolled cold. At that date the rolling was done so cold in each pass that the light "trains" would break frequently in rolling the rails. To prevent this, the steel was worked and finished at higher temperatures, in short time. Specimens of these rails have been sent to the U. S. Arsenal at Watertown for complete physical tests, and the carbons are to be determined by combustion, to see what they actually were. The mill test of the carbons for technical investigations is not





No. 10. 100 lb. Sec., N. Y. C. & H. R. R. R., L. I. & S. Co., 1892.  
Magnified 50 diameters. 14 inch sq. Ingot on Base.



No. 11. 100 lb. Sec., N. Y. C. & H. R. R. R., L. I. & S. Co., 1892  
Slag in centre of cut. Magnified 50 diameters.  
4,000 Granulations per sq. inch. 14 inch sq. Ingot on Base,



sufficiently precise, as it is combined in such a way that the color test does not show what it was, when the steel is rolled so cold.

The microstructure of the Barrow and the Griswold steel is interesting, from the fact that they were made from small hammered ingots and rolled cold. Part of this structure is due to the mechanical work from the hammer and the rolls. If these pieces of steel are reheated up to about  $800^{\circ}$  C. this structure disappears, and a fine granular structure takes its place, and it is not possible to reconstruct the original structure as found in the rails by any method of heat treatment known at present.

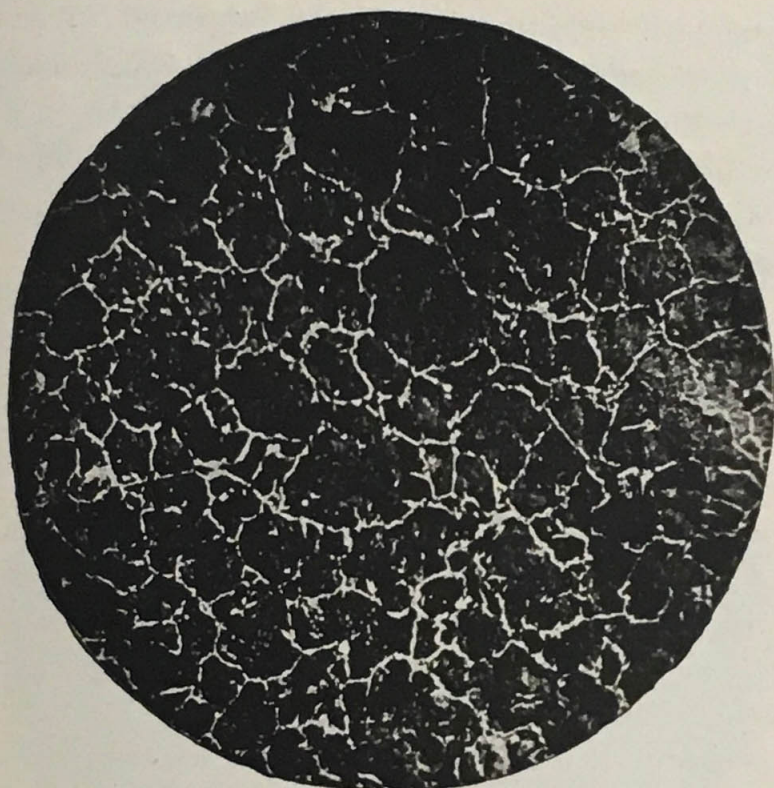
This feature of the structure of the steel has not been investigated to any extent. The difficulties are not only great, but very expensive, to reproduce the structure by the combined conditions incident to the manufacture of the early rails. Forging may reproduce this structure, at little expense, but so far has not proved satisfactory.

I have sent a number of these old sections, — and it is important to collect more of the old rails which have been in service, — to Watertown, to the U. S. Government Testing Machine, to obtain complete physical tests of the early steel rails. One important fact is well established, — that with their moderate elastic limits in the metal of their sections, they did not remain smooth in the track, but took permanent sets, so that the track could not be maintained in a smooth condition by the trackmen.

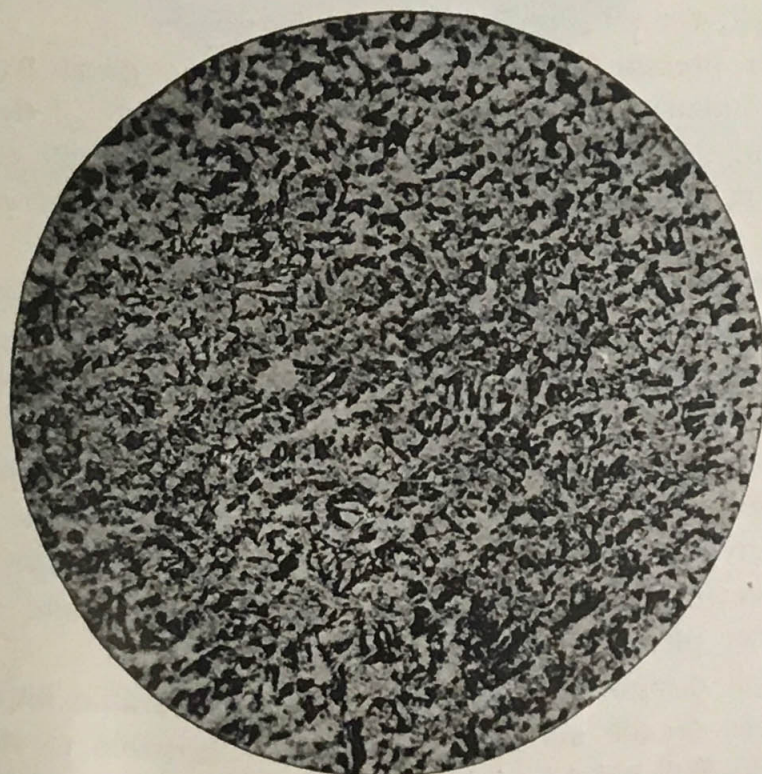
While the stiffness of recent sections has been increased 60, 80, and over 100 per cent, the axle load effects have increased in a faster ratio, and rails of higher elastic limits are needed for present service.

The hammering of the early ingots actually welded a great many of the minute blow holes which were in the low carbon steel, and by work being continued at a low temperature, an excellent wearing structure was secured. But to attempt to repeat this structure in our present rails, without taking into consideration the elastic limits, as is done generally by engineers, is to make a very serious mistake. The physical properties of the steel were too low for girders. It is unnecessary for me to call your attention to the fact that in order to have the





No. 12. Coarse Granular. 64 lb. Sec., N. Y. C. & H. R. R. R., Wilson Cammel, England, 1870. Magnified 50 diameters. 3,600 Granulations per sq. inch.



No. 13. Non-Granular, 65 lb. Sec., N. Y. C. & H. R. R. R. Barrow Steel, England, 1873. Magnified 50 diameters. 10,000 Incomplete Granulations per sq. inch.



requisite standard of smoothness now required in your track, the metal of the rails must have sufficiently high elastic limits to withstand the strains of the present locomotives expending their large tractive effort, without taking a set. The work of the locomotive will increase, year by year, as well as some additional weight of axle loads.

The consumer can determine better what physical properties are essential in the rails, to withstand the service, than those who give the subject superficial examination. Many who are now advocating colder rolling of the rails, do not take into consideration what physical properties will be obtained in the steel.

The whole subject, with most people, seems to be a question of wear of the steel. While this is important, it is necessary to have the requisite physical properties in the rails, to enable them to sustain the loads as girders, without taking permanent sets in the track under the traffic.

The problem, or series of problems, of making a rail adapted to the present service, is not as simple as many seem to think, either in a technical or commercial sense. It will require the coöperation of railway officials to secure what is really needed in the track, for the present and future service.

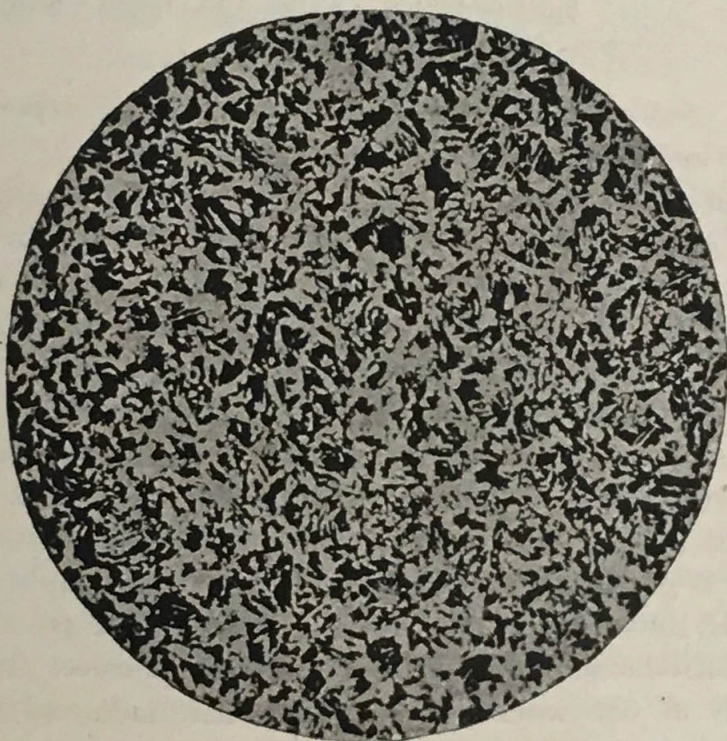
Under present locomotives, expending so great tractive efforts, the intensity of pressure between the treads of the driving wheels and the surface of the rails, is from  $2\frac{1}{2}$  to 3 times as great as on the early steel rails, which were considered to wear so well.

Tires are wearing more rapidly than under the lighter wheel loads and service, and in some instances irregular deformations of the circumference have occurred, by detrusion of metal in the treads of tires.

In the rails as girders, we can distribute the wheel loads by stiffness of the sections, but we must sustain the intensity of the driving-wheel load pressures by only a slight increase of the bearing surfaces, of width of rail head, and combined with higher physical properties in the steel.

In roll designing, there has been considerable improvement in the past decade and I find that it is possible to start a set of rolls the full height of the section, when new, and by careful dressing of the intermediate rolls, the height can be maintained practically the same for the different rollings. I am opposed

decidedly to allowing  $\frac{1}{32}$  over and  $\frac{1}{32}$  under the height of the section for dressing the rolls. I think if the railroads insist upon it, the height of the rail can be kept practically that of the section, without increasing the cost to the mill, but care would be



No. 14. Non-Granular, 65 lb. Sec., N. Y. C. & H. R. R. R.  
John Griswold steel, Troy, 1874. Magnified 50 diameters.  
12,000 Incomplete Granulations per sq. inch.

required in dressing the rolls. A description of the means necessary to do this is far too technical for this report, and it would need entire drawings of the rolls to show exactly what I mean; but I have no difficulty in practically maintaining the height of the section for several rollings.



## THE TAYLOR-WHITE PROCESS OF TREATING TOOL-STEEL \*

[Being the Report of the Committee on the Invention of Maunsel White and Fred W. Taylor. Sub-Committee: Charles Day, James Christie, Coleman Sellers, Arthur Falkenau, Wilfred Lewis.]

**Y**OUR Sub-Committee submits the following report on the Taylor-White process of treating tool-steel.

About three years ago an extensive series of experiments were undertaken at the Bethlehem Steel Works, by Messrs. Taylor & White, in order to determine the relative efficiency of various brands of tool-steel on the market at that time.

There are two distinct classes of tool-steel, namely, carbon and air or self-hardening. The latter brand, the result of Mushet's work, has completely replaced the carbon steel for roughing, its comparative efficiency approximately being 1.5 to 1.0. Mushet discovered that by the addition of manganese and tungsten to tool-steel it maintained its cutting edge at much higher temperatures and consequently much higher speeds were possible. The general introduction of this steel did not, however, take place as rapidly as one would suppose, the manufacturers failing to appreciate the great economy realized by using it. In fact, very few of the shops that did use it obtained the greatest efficiency possible, as no knowledge of cutting speeds and feeds was at hand.

The object of the experiments above referred to was largely to obtain this data for different materials and to determine the tool best adapted to their work.

It was found that the results obtained from different tools made from the same steel varied greatly, and as the only way of accounting for it was by variations in the process of hardening, it was decided to make a thorough investigation along these lines.

Having decided upon a brand of steel, tools were forged and heated to different temperatures and then put in a lathe and tested. It was found that those cooled from a very high temperature gave remarkable results far surpassing anything yet accomplished.

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\* Journal of the Franklin Institute, February, 1903.



It must be understood at this point, that all air-hardening steel manufactured up to this time, was hardened by heating to a cherry red and either allowed to cool gradually or in a blast of air. Users were invariably cautioned against overheating, cherry red being specified as the desired temperature.

The above tests marked the beginning of a most thorough series of experiments, the object being to determine the best chemical composition and temperature for treating. In carrying out the above experiments a 66-inch Bement-Miles lathe was belted to an Evans friction-cone countershaft, it in turn being geared to a 40 horse-power Westinghouse motor. By this means any desired speed could be obtained. A depth of cut of  $\frac{3}{16}$  inch and feed of  $\frac{1}{16}$  inch with a duration of test of twenty minutes was adopted as standard. Two hundred tons of forgings were cut up in carrying out this work, the total cost aggregating \$100,000.

A review of the patent specifications may now be well in order that we understand exactly the claims made. Messrs. Taylor & White say in part: "Our invention relates to the manufacture of tools for cutting metals or similar uses where the tool is highly heated in performing its work, the object of our invention being to provide a tool capable of working at higher temperature, and consequently doing more work in a given time than the tools as heretofore made." The above statements make it very clear that the tool is adapted to *roughing work only*, and unless sufficient speed is obtained, no gain in output can be had. We wish to make this point perfectly clear.

Again, on the first page of the patent papers (Pars. 65 - 90). we find a general statement of the invention which may be worth quoting: "Our invention is based on our discovery that, while it is true tools made of air-hardening steels all deteriorate at temperatures in excess of a bright cherry red (though it must be understood, not all at the same temperatures), it is also true that when air-hardening steels are made with certain constituents in ascertained proportions this deterioration only prevails during a limited range of temperatures above the bright cherry red; that is to say, from about 1,550° F. to about 1,700° F. (corresponding to a light salmon color), and on our further discovery that above this range of temperature, which we will call the "breaking-down point," and from 1,725° F. up to a tem-



perature at which the steel softens or crumbles when touched with a rod (approximately 1,900° or 2,000° F.) the efficiency of tools of such special steels, that is to say, their cutting speeds and also their uniformity in efficiency, is greatly increased and largely so in proportion to the degree of heat to which they are raised. This is so much the case that their cutting speed may be stated to be from one and a half to two and a half times that

Link-Belt Engineering Company, Nicetown, Phila.

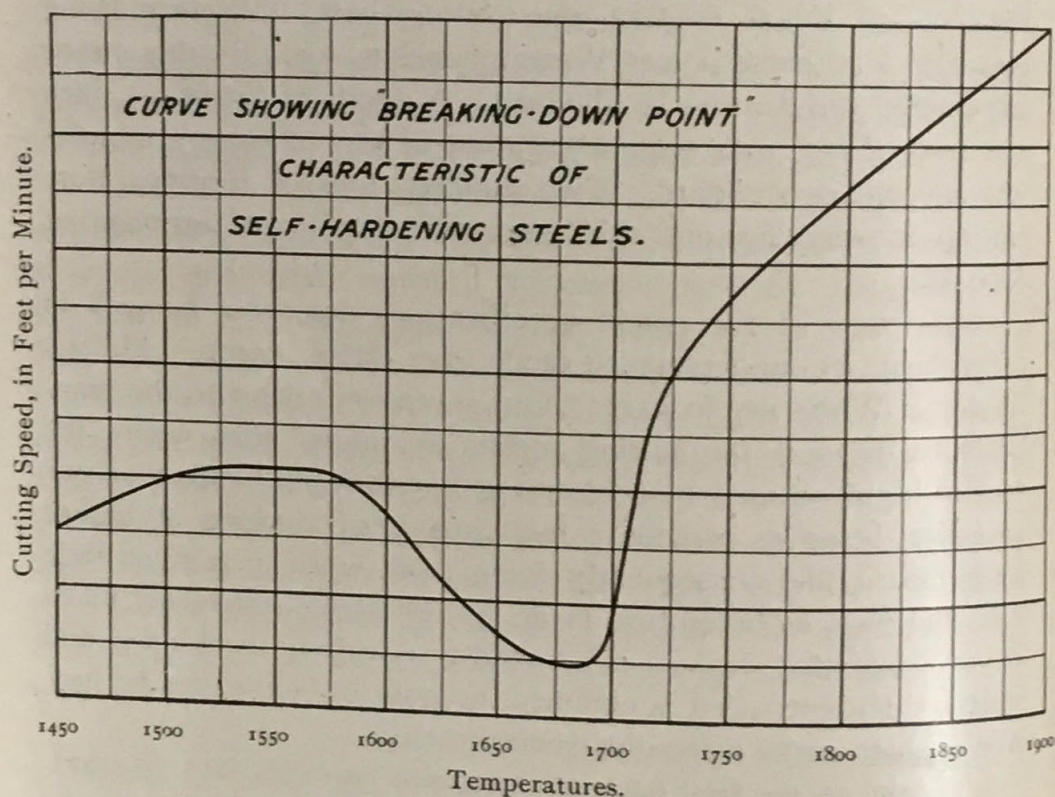


Fig. 1.

of the tool heated as heretofore, to a temperature below the breaking-down point. The accompanying curve of temperature and cutting speed shows very clearly in a graphical manner the principle of the Taylor-White discovery, and also the reason why a cherry red was deemed best heretofore, as the curve falls off at this point.

How true the above statement of increased efficiency is, can be judged later when considering the experiments made recently by the Sub-Committee.

In order to obtain a suitable steel for the Taylor-White



treatment it is necessary that it should be compounded with chromium in the proportion of at least  $\frac{1}{2}$  of 1 per cent, and another or other members of the commercially available members of the chromium group of metals in the proportion of at least 1 per cent; that is to say, with either tungsten or molybdenum in the proportion of at least 1 per cent. They also state that materially better results are obtained in some cases by increasing these proportions. The relative value of the various members of the chromium group is carefully considered in the patent papers, and it is interesting to note that the percentage of carbon seems to have little or no effect on the results, steels containing from 85° C. to 200° C. having the same efficiency.

We naturally ask, what changes does the steel undergo when subjected to this high heat? and in answer will again quote from the patent papers, page 2, paragraph 50: "We do not feel able to state with certainty the chemical and molecular changes which occur in steels of this composition when heated above the breaking-down point, and to the high-heat characteristic of our process, but will mention as one characteristic change due to our treatment, that the tools, after exposure to the high heat, show by analysis a *smaller* percentage of carbide of chromium than existed in the steels before such treatment; for example, in steels containing 1.50 per cent chromium and 0.7 per cent carbide of chromium, the tool after treatment contained but 0.2 per cent of carbide of chromium, and in steels containing 0.75 per cent of chromium and 0.9 of carbide of chromium, the treated steel contained but 0.3 per cent of carbide of chromium.

Again, air-hardening steels of this composition, like air-hardening steels in general, possess in their normal condition the characteristically fine velvety grain when fractured. The higher range of temperature necessary in our treatment has a very noticeable tendency to change the structure of the metal and to give a non-velvety appearance and coarser grain, frequently interspersed with sparkling grains. "When treated with the higher heats and to obtain the best results, the steel of the tools shows under the microscope a distinctly large-grained structure, in many cases intercepted with austenite or microconstituent of steel discovered by Osmond, the chemical composition of which is unknown, and which, according to the best authorities, has never been met with in the industrial treatment of steel."



The question of cooling the tool is considered in detail, different methods being adopted for various duties. They may be in general stated, however, as follows: The tool is cooled rapidly from the high heat to a point below the breaking-down temperature in a lead bath, then slowly in the air or lime, etc., as the

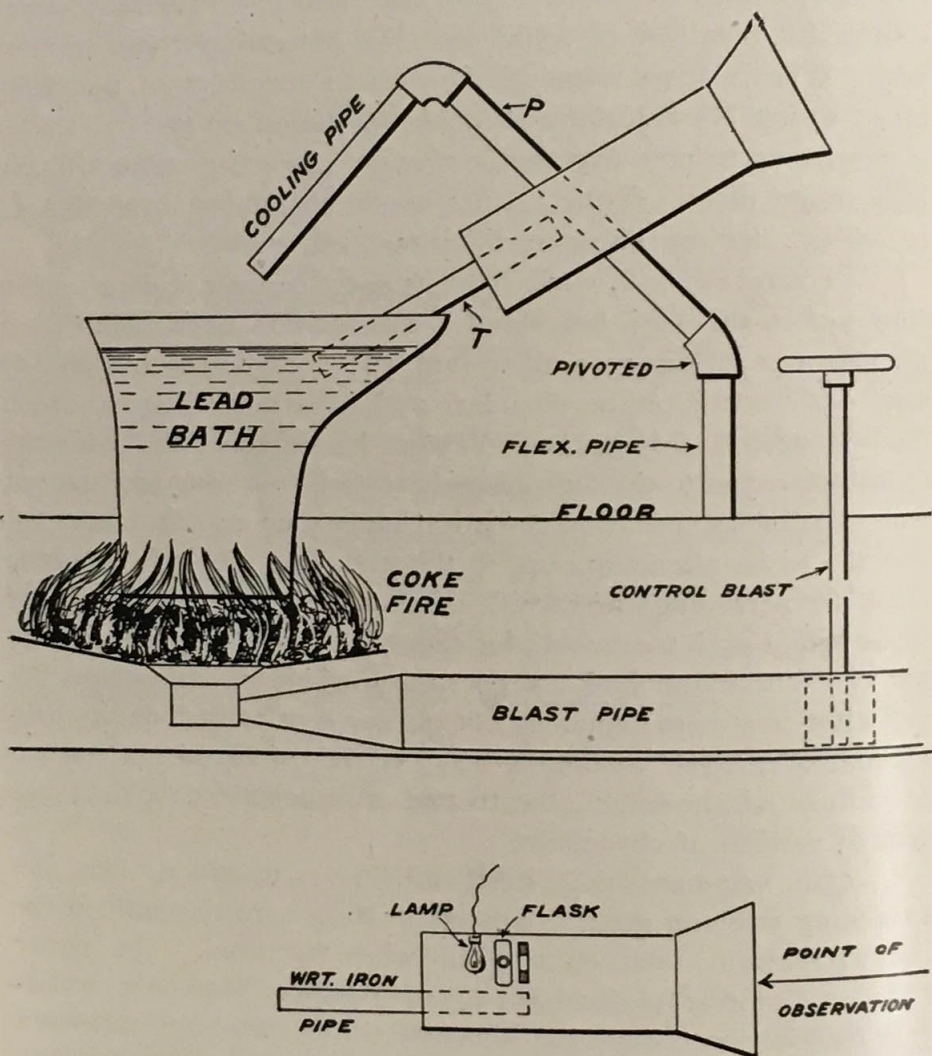


Fig. 2.

case may be. It is very essential that at no time the temperature should rise, as in such a case the tool would be seriously impaired. After the tool has cooled off, its efficiency is found to be further increased by subjecting it to what is termed the low heat for about ten minutes, this temperature ranging from  $700^{\circ}$  F. to  $1,200^{\circ}$  F. One of the chief claims made by Messrs. Taylor &



White is the great uniformity obtained by their process, which makes it possible to run every tool to a very high efficiency. This uniformity is obtained by the apparatus employed, by means of which remarkable close temperatures can be ascertained. The tool, after being forged, is placed in a coke furnace, where it is heated gradually to a high heat, the latter being designated as the point at which the steel crumbles when tapped with a rod. As the tool is incandescent at this point, it is necessary that the operator wear colored glasses while testing for heat.

After heating, the tool is rapidly drawn from the furnace and plunged into a lead bath, which is maintained at a certain temperature by a very ingenious method; a sketch may help to make this part of the process more clear. The cast-iron retort shown is heated from below by means of a coke fire, the intensity of which may be increased at will by means of a blast of air. If the temperature rises too high, the closed pipe *P*, which has cold water circulating in it, can be lowered into the bath. The temperatures are determined as follows: From a lip in one side of the crucible is placed a pipe *T*, which projects into the lead bath. This pipe is enclosed in turn in a wooden tube blackened inside, also containing a flask of fluid lighted by an incandescent lamp in such a position that its color may be compared directly with the bath as seen through pipe *T*. Great care has been exercised in obtaining chemical ingredients for different temperatures that would be uniform, and of course the lamps must be standardized and voltage kept constant by means of a rheostat. It must be thoroughly understood that it is in no way essential to use apparatus of this character to obtain a treated tool. In fact, your Committee has seen very good results obtained from tools treated in an ordinary smithshop fire and cooled in a blast of air. We do not feel, however, that uniformity can be had by the latter method, and as it is this point that makes the treatment so valuable, we think that the great stress should be laid on the apparatus. The tools at Bethlehem are treated by laborers, who receive \$1.35 per day. It might be well to mention at this point that in some cases where it is desirable to grind the tool before treating, a flux is used in order to prevent burning at the high heat.

In order to verify the statements made in the patent papers with regard to increased efficiency at high speeds, and to investigate the apparatus above referred to, your Committee visited



Tool-Steel Tested	Date	Exp. No.	Forging No.	Feed	Depth of Cut	Cutting Speed Aimed at	Duration of Cut	Average Cutting Speed Obtained	Condition of Tool at End of Run	Remarks
				inch.	inch.					inch.
T.W.H. 2313	Dec. 19	1	7238 C <sub>1</sub>	$\frac{1}{16}$	$\frac{3}{16}$	11	20	11	Fair	$5\frac{7}{8}$ travel
Mushet . . . .	"	2	"	$\frac{1}{16}$	$\frac{3}{16}$	5	(*13 $\frac{1}{2}$ )	5	Point gone completely	$1\frac{15}{16}$ "
"	"	3	"	$\frac{1}{16}$	$\frac{3}{16}$	3 $\frac{1}{2}$	(†12 $\frac{1}{2}$ )	3 $\frac{1}{2}$	"	$1\frac{1}{2}$ "
Sanderson . .	"	4	"	$\frac{1}{16}$	$\frac{3}{16}$	3 $\frac{1}{2}$	20	3 $\frac{1}{2}$	gone	$1\frac{7}{8}$ "
Boreas . . . .	"	5	"	$\frac{1}{16}$	$\frac{3}{16}$	4 $\frac{1}{2}$	20	4 $\frac{1}{2}$	"	2 "
Mushet . . . .	"	16	"	$\frac{1}{16}$	$\frac{3}{16}$	3	4	3	"	"
Raeburn . . .	"	17	"	$\frac{1}{16}$	$\frac{3}{16}$	3	20	3	fair	$1\frac{3}{4}$ "
Mushet . . . .	"	18	"	$\frac{1}{16}$	$\frac{3}{16}$	3	20	3	gone	$1\frac{3}{4}$ "
T.W.M.E. 2806	Dec. 20	6	19788 B <sub>1</sub>	$\frac{1}{16}$	$\frac{3}{16}$	140	20	140	good	$39\frac{3}{4}$ "
Sanderson . .	"	8	"	$\frac{1}{16}$	$\frac{3}{16}$	72	20	72	fair	$19\frac{3}{8}$ "
Mushet . . . .	"	9	"	$\frac{1}{16}$	$\frac{3}{16}$	72	1 $\frac{3}{4}$	72	gone	$1\frac{3}{4}$ "
Boreas . . . .	"	10	"	$\frac{1}{16}$	$\frac{3}{16}$	72	6 $\frac{1}{2}$	72	"	$6\frac{3}{8}$ "
Mushet . . . .	"	11	"	$\frac{1}{16}$	$\frac{3}{16}$	60	19	60	"	$15\frac{3}{4}$ "
Sanderson . .	"	17	"	$\frac{1}{16}$	$\frac{3}{16}$	82	19 $\frac{1}{4}$	82	"	$22\frac{1}{2}$ "
Benj. Atha . .	"	13	"	$\frac{1}{16}$	$\frac{3}{16}$	82	13	82	"	$15\frac{1}{4}$ "
T.W.M.E. 2757	"	14	"	$\frac{1}{16}$	$\frac{3}{16}$	156	20	156	good	$40\frac{1}{8}\frac{1}{2}$ "
T.W.B. 0.3 . .	"	19	1506 A	$\frac{1}{16}$	$\frac{3}{16}$	70	20	70	gone	$16\frac{7}{16}$ "
Boreas . . . .	"	20	"	$\frac{1}{16}$	$\frac{3}{16}$	55	20	55	good	$12\frac{3}{4}$ "
"	"	21	"	$\frac{1}{16}$	$\frac{3}{16}$	60	38	—	gone	$2\frac{9}{16}$ "
Mushet. . . .	"	22	"	$\frac{1}{16}$	$\frac{3}{16}$	50	20	50	"	$11\frac{9}{16}$ "
Benj. Atha . .	"	23	"	$\frac{1}{16}$	$\frac{3}{16}$	50	20	50	good	$11\frac{5}{8}$ "
T.W.B. 0.3 . .	"	24	"	$\frac{1}{16}$	$\frac{3}{16}$	70	20	70	"	$16\frac{1}{2}$ "
Benj. Atha . .	"	25	"	$\frac{1}{16}$	$\frac{3}{16}$	55	3 $\frac{1}{2}$	—	gone	$2\frac{3}{16}$ "

\* Examination of tool showed that it failed long before end of test.

† Point of tool red-hot for 13 minutes.

‡ Point of tool red-hot for 15 minutes.

#### Chemical Composition of Above Forgings

	Bar No. 7238 C'	Bar No. 19788 B'	No. 1506 A
			Total 3.854
Carbon . . . .	.876	.105	Graphite 2.948
			Combined .906
Manganese . . .	.62	.36	.43
Silicon . . . .	.24	.025	.98
Phosphorus . . .	.025	.023	.298
Sulphur . . . .	.028	.035	.055

#### Angles of Tools Tested

	Hard Bar.	Soft Bar.	Cast-Iron.
Clearance . . . .	9°	6°	6°
Front rake . . .	5°	8°	14°
Side rake . . . .	9°	12°	18°



the Bethlehem Steel Works on December 20 and 21 in 1901. Various brands of air-hardening steel were purchased and hardened in the presence of the Committee, according to the manufacturers' instructions. These tools were forwarded to the Bethlehem and ground to the standard angles as given below. The tests were primarily made out at Bethlehem on account of the experimental lathe above referred to, it being the only one where any desired speed could be obtained with sufficient power to pull the cut. Three different test-pieces were experimented on—two of steel and one of cast-iron. All the tools that were tested were brands that were on the market at the time the Taylor-White patents were granted.

Numerous tests have been made before and published, but none that we had access to showed the relative value of various tools in the true light; consequently we decided to run the tools at the same depth of cut and feed, varying the speed in order that each tool should last twenty minutes. It will be seen at once that an exhaustive test of this kind would take a very long time, as the speed must first be approximated many times before the right one can be obtained. The consequence was that the tests were not as full as we had desired, but we feel they are sufficiently convincing for our purpose.

The table on p. 136 shows the data just as we obtained it, an explanation of forgings, numbers, etc., being given below. It must be understood that all the tools compared with the Taylor-White in these tests were tools that were on the market at the time of the invention. We will consider later various treatments that have come into existence since the Taylor-White patents were granted.

An examination of the tests on the 86° C. hammered forging shows that the relative efficiency of the treated tool and best untreated tool is 11 to 3 or more than 3.5 to 1, for the soft forging (10 carbon) 156 to 70 or 2.2 to 1, and for the cast iron 70 to 55 or slightly less than 1.3 to 1. These figures show that for steel the efficiency is much greater for hard forgings than for soft, but even in the latter case exceeds two to one, while as we might expect, the saving on cast-iron is much less, being about 1.25 to 1. On hard castings, however, the gain is much more, often reaching 2 to 1, and on this account it is well adapted to certain work. In proof of the latter statement we might add that



at the Link-Belt Engineering Company's Works, Nicetown, hard-sprocket wheels can be bored at more than double the speed with Taylor-White tools.

There were many points of interest in the above tests for any one who was not familiar with the operation of treated tools working on low-carbon steel. Reference to the table shows a cutting speed of 156 feet per minute, with  $\frac{3}{16}$  inch depth of cut and  $\frac{1}{16}$  inch feed. This tool was removing metal at the rate of 353 pounds per hour and was *red-hot*  $\frac{5}{16}$  inch from the point. The color was distinctly visible in the daylight, no stronger proof being needed of the high heat at which these tools maintain their cutting edge. At the end of twenty minutes the edge of the tool was carefully examined with a magnifying glass and found to be *perfect*. In fact, the original grinding-marks could still be detected; consequently, we see that the claims made by the inventors regarding increased efficiency for roughing work are just, so far as the brands of steel we compared are concerned. The importance of this discovery can scarcely be overestimated when we consider how its influence is felt in every establishment where cutting tools are used.

If the manufacturer is doing a large proportion of roughing, it may be the means of doubling his output, assuming that he has already been using the best-known steel. The discoveries of Messrs. Taylor & White, coming, as they did, at a most opportune time, have been an epoch in machine practice, and have simply proved again that scientific methods lead to much better results than guess-work. The great interest which their work awakened in the engineering world was largely due to Mr. Taylor's system of controlling the men at Bethlehem Steel Works, which made it possible to run every machine to a much higher efficiency than has heretofore been thought of. We also feel that the Taylor-White steel has done more for a certain class of machine design than any other thing we can point to. It is no longer desirable to have close-speed regulation, etc., but absolutely essential if maximum output is desired. The entire problem has become one of scientific study instead of guess-work, and it is to such work as that followed out by Messrs. Taylor & White that we must look in the future.

In another part of this report reference was made to treatments which have been put on the market since the Taylor-White



patents were issued. Several manufacturers of tool-steel claim to have a tool that gives results quite equal to the Taylor-White, and it was our desire to have such tools entered in the above test.

As several of the manufacturers object to making them at the Bethlehem Steel Works, it was deemed best to postpone these trials until a later date, when they can be made at the Link-Belt Engineering Company on a lathe especially adapted to this work and which they expect to have in operation in a few weeks. If such a series of tests are made it will, of course, be necessary to prove that the various tools do not infringe the patents we are considering.

In conclusion, your sub-committee takes pleasure in recommending the award of the Elliott-Cresson Medal to Messrs. Taylor & White for the discovery and development of a method of treating a certain composition of tool-steel which has made it possible to largely increase the output of machines doing roughing work.

Adopted April 11, 1902.

Attest:

WM. H. WAHL,  
Secretary.

## PROBABLE EXISTENCE OF A NEW CARBIDE OF IRON, $\text{Fe}_2\text{C}$ .\*

By E. D. CAMPBELL and M. B. KENNEDY

THE only well-recognized carbide of iron which has been recovered from specimens of commercial iron or steel is that which was first described by Sir F. Abel and Mr. Deering in 1885.† This carbide, according to their figures, is represented by the formula  $\text{Fe}_3\text{C}$ . Abel and Deering's work has been repeated since, with various modifications and improvements in method, by Müller,‡ Osmond and Werth,§ Arnold and Read,|| and by one of the present authors.¶ The results obtained by all these experimenters confirm the conclusion drawn from the work of Sir F. Abel, that the greater part of the carbon in annealed steel exists

\* *Journal Iron and Steel Institute*, 1902, II.

† *Proceedings of the Institution of Mechanical Engineers*, 1885, p. 30.

‡ *Stahl und Eisen*, Vol. V. § *Annales de Mines*, 1885, Vol. II.

|| *Journal of the Chemical Society*, 1894, Vol. LXV, p. 788.

¶ *American Chemical Journal*, 1896, Vol. XVIII, p. 836.



in the form of a definite carbide having the empirical formula  $\text{Fe}_3\text{C}$ . The carbide obtained by all the authors mentioned has been recovered from steel containing, in most cases, less than 1 per cent of carbon. Perhaps the most exhaustive research is that of Arnold and Read, who show that, even in carefully annealed steel, only about 72-94.9 per cent of the total carbon in the metal is recovered in the form of carbide. The failure to recover all of the carbon as pure carbide seems to be due to the decomposition of carbide of iron, not crystallized or precipitated, but remaining in solid solution. As might be expected, carbide of iron, held in solid solution in the iron, would be much more easily decomposed by the action of dilute acids than if the carbide were precipitated or crystallized in masses of appreciable size. If we consider the conditions under which the carbide recovered by Arnold and Read was formed, we may readily account for the failure to recover the total carbon of the steel in the form of carbide. When the metal, being annealed, is above the recalescent point  $Ar_1$ , carbon exists, according to different authors, in one of the following forms: as carbide of iron in solid solution, as a sub-carbide of iron, or as carbon dissolved in an allotropic form of iron. When the recalescent point is reached one of the following changes would take place, according to the view held as to the condition of the carbon at the higher temperature; the dissolved carbide of iron precipitates or crystallizes from solid solution, the subcarbide of iron splits up or separates into the normal carbide; or, according to the third view, the dissolved carbon combines with iron to form the definite carbide  $\text{Fe}_3\text{C}$ . In any of these cases we have the conversion of martensite into pearlite during the recalescence, and most of the carbon passing into the form of pure carbide  $\text{Fe}_3\text{C}$ , which constitutes the bright laminae of pearlite. It would seem almost inevitable that the portion of pearlite colored dark in the preparation of specimens for microscopic examination must consist of a solid solution of carbide of iron in iron, and not pure iron, or ferrite, as is usually assumed. The decomposition of this dissolved carbide of iron would readily account for the failure to recover all of the carbon in steel in the form of precipitated or crystallized definite carbide  $\text{Fe}_3\text{C}$ . The idea that the inability to recover all the carbon of annealed steel in the form of pure carbide is due to the decomposition of carbide remaining in solid solution is supported by the results obtained by Arnold and Read in the treat-



ment of hardened steel. Their results seem to show that the carbides in solid solution are very much more readily decomposed than when precipitated or crystallized. The fact that they obtained 20-38 per cent of the total carbon as the carbide  $\text{Fe}_3\text{C}$ , however, would tend to show that at the critical point  $\text{Ac}_1$ , the carbides of iron merely pass into solid solution, perhaps with partial, but certainly not complete, dissociation into iron and dissolved carbon.

Since it has been so well demonstrated that the carbide of iron separating from solid solution in the presence of a very large excess of iron has the empirical formula  $\text{Fe}_3\text{C}$ , the question arose in our mind as to whether a carbide crystallizing or precipitating from solid solution in the presence of a much larger proportion of carbon than is found in ordinary steels might not have a different composition. In order to decide this question, we have isolated the carbide from a sample of annealed white iron. The iron employed for this purpose was a pig of white charcoal iron, furnished us through the courtesy of Mr. L. E. Dunham, manager of the Ashland Iron and Steel Company, Ashland, Wisconsin. The original iron had the following composition: combined carbon, 3.53 per cent; graphite carbon, 0.01 per cent; silicon, 0.07 per cent; phosphorus, 0.13 per cent; sulphur, 0.01 per cent; and manganese, 0.05 per cent. The pig was broken up, and a piece weighing about 900 grams selected for the work. This piece was first dressed with an emery wheel until perfectly bright, and was then placed in a porcelain evaporating dish, covered in turn with a second dish, so arranged that a stream of hydrogen could be kept up during the annealing. The temperature to which the piece was raised was measured by means of a Le Chatelier thermocouple placed close to the iron. The dishes containing the iron were placed in an assay furnace, and the temperature gradually raised during a period of five hours to  $950^\circ\text{C}$ . After maintaining the iron at a temperature varying from  $930^\circ\text{C}$ - $950^\circ\text{C}$ . for about two hours, the temperature was allowed to fall, about three hours being required for it to reach  $600^\circ$ . An analysis of the iron so annealed showed that the graphite carbon had increased only to 0.12 per cent. This piece of annealed metal, supported by means of a heavy copper wire, was connected with the anode of a storage battery, the cathodes, one on each side of the bar, being platinum cylinders. The solution used as an electrolyte was fourth-normal



sulphuric acid, about three-fourths of the iron being immersed during the electrolysis, which was conducted in a large beaker containing about four litres. In the electrolysis, an electromotive force of two volts and a current of 0.30 ampere was found to give the most satisfactory results. When such a current as this had acted for twenty-four hours, the iron was taken down and the adhering carbide rubbed loose under water by means of a brush made of very fine aluminium wire, such as employed by one of us in the recovery of pure carbide of iron, referred to in the first part of this article. When the carbide had been removed, fresh acid was placed in the beaker and the electrolysis continued. The recovered carbide was first triturated lightly under water and washed by decantation with water until, on stirring, no light carbonaceous matter could be seen carried over with the water. The water was then decanted off and the carbide treated with a cold 20 per cent solution of KOH in order to dissolve small pieces of aluminium wire. When the aluminium was completely dissolved, the carbide was again thoroughly washed by decantation with water, and was finally rinsed on to a smooth, hardened filter-paper, and washed with alcohol followed by ether. This carbide was then placed in a large test-tube, and dried at a temperature of about  $250^{\circ}$  in a stream of pure, dry hydrogen. The weight of the original iron was taken before the electrolyses began, and after each two or three electrolyses the bar was again weighed to determine its loss, and the corresponding carbides weighed and analysed. The results of these electrolyses will be best shown in the following table.

TABLE I

Recovery	Loss in Weight of Bar	Weight Carbide Recovered	Per cent C in Carbide	Per cent Fe in Carbide	Total	Weight C in Carbide	Weight Fe in Carbide
1-2-3	30.68	1.279	6.53	93.90	100.43	0.0835	1.2009
4-5-6	19.02	0.715	6.52	94.46	100.98	0.0466	0.6753
7-8-9	75.45	28.192	8.68	90.23	98.91	2.4470	25.4376
10-11-12	24.85	7.083	6.99	92.30	99.29	0.4951	6.5376
13-14	23.10	6.854	7.07	93.12	100.19	0.4845	6.3824
15-16	35.60	23.602	7.86	91.08	98.94	1.8551	21.4967
17-18-19	39.29	17.170	8.49	90.20	98.69	1.4577	15.4873
20-21	21.11	8.605	7.72	90.09	97.81	0.6643	7.7522
22	32.95	4.839	9.45	89.40	98.85	0.4572	4.3260
23-24		7.805	7.95	91.03	98.95	0.6204	7.1048
Total	302.05	106.144				8.6114	96.4008

302.05 grams of iron with 3.53 per cent carbon would contain 10.662 grams of carbon; the amount of carbon recovered in the carbides, 8.6114 grams, is therefore 80.76 per cent of the total. If we calculate the carbon and iron only, recovered as carbide, we would have an average composition of carbon 8.20 per cent and iron 91.80 per cent. The irregularity in the proportion of carbide recovered, to loss of weight of the bar, is due to the irregular dissolving away of the iron during the different electrolyses. Some days a small yield of carbide would be obtained in proportion to the weight of metal dissolved, and perhaps the next day a comparatively large layer of carbide would be removed. At the end of all the work the piece of iron was found to be covered with a rather firmly adhering, but porous layer, which could be scraped off to a depth of nearly a millimetre, if considerable pressure was applied. This was not, however, included in the above table. The carbides recovered had in general a gray metallic appearance when examined in mass with the naked eye, some samples being brighter than others. Under the microscope it appears, as would be expected from a substance precipitating or crystallizing from solid solution, "an amorphous black powder, without apparent crystalline form." Sample No. 22, when first recovered, seemed to be the brightest of any, and was kept separate for this reason before an analysis of it was made. These carbides were all in the form of a powder fine enough to easily pass a sieve with 100 meshes to the linear inch, but a considerable portion would be retained by a 200 mesh sieve. Three of the samples of the carbide were separated by means of a 200 mesh sieve into two portions, coarse and fine. The results of these separations, with the analyses of the separated portions, are given in the following table:

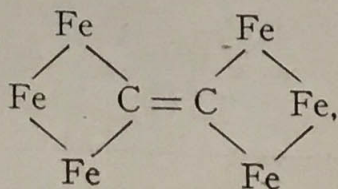
TABLE II

Sample	Per Cent Coarse	Per Cent Fine	Carbon in Coarse	Carbon in Fine
7-8-9	36.01	63.99	7.51	9.31
20-21	52.76	47.24	6.94	8.80
22	38.46	61.54	8.76	9.87

A determination of the graphitic carbon in the fine material from sample 22 gave only 0.12 per cent. Those samples con-

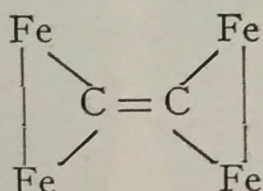


taining the highest per cent of carbon do not seem to have been decomposed by the acid in the process of recovery to any greater extent than the samples with the lower per cent of carbon. This fact, taken together with the fact that all the samples were thoroughly washed by stirring and decantation, would seem to prove that the carbon in these carbides is actually chemically combined with the iron, and not mechanically mixed as carbonaceous matter resulting from chemical decomposition. Pure carbide of iron  $\text{Fe}_3\text{C}$  contains 6.67 per cent carbon, while a carbide  $\text{Fe}_2\text{C}$  would contain 9.67 per cent. It will be noticed that within the limits of experimental error, the carbon percentages range between that of  $\text{Fe}_3\text{C}$  and that of  $\text{Fe}_2\text{C}$ . It does not seem as likely that iron would form a large number of carbides with the percentage of carbon varying between 6.67 per cent and 9.67 per cent, as that it would form two carbides, one having the empirical formula  $\text{Fe}_3\text{C}$ , and the other  $\text{Fe}_2\text{C}$ . The most probable explanation of the results obtained here is that we have obtained varying mixtures of the two above-mentioned carbides. The chemical behavior of these two carbides is almost identical. The  $\text{Fe}_2\text{C}$  seems to be more hard to dissolve in potassium cupric chloride and in hydrochloric acid than is the  $\text{Fe}_3\text{C}$ , but the difference is not sufficiently marked to be used as a means of quantitative separation. The hydrocarbons given off on dissolving the carbides in hydrochloric acid are practically the same, qualitatively and quantitatively. About one-half of the gaseous hydrocarbons are paraffins and one-half olefins, yielding from 3.1 to 3.3 times their own volume of carbonic anhydride on explosion. The high molecular weight of the products of solution would indicate that the molecular formulæ of the carbides are not shown by the formulæ  $\text{Fe}_3\text{C}$  and  $\text{Fe}_2\text{C}$ , but more probably by some multiple of these formulæ, as has been expressed by one of us in discussing the constitution of steel.\* If the constitution of the ordinary carbide of iron is represented by the general formula  $(\text{Fe}_3\text{C})_n$  and the structure of one of the lowest ferro-carbons by the formula —





it is conceivable that one Fe atom might be removed from each  $\text{Fe}_3$  group, leaving the two remaining Fe atoms in the same relation to the carbon atoms as they bore in the original ferro-carbon. Such a carbide would have a general formula  $(\text{Fe}_2\text{C})_n$ , and the structure of the derivative from the above member would be —



Carbides of the general formula  $(\text{Fe}_2\text{C})_n$  derived in this way from carbides of the general formula  $(\text{Fe}_3\text{C})_n$  would give the same products of solution in acids. The fact that  $(\text{Fe}_2\text{C})_n$  is recovered only from iron containing a large percentage of carbon, while  $(\text{Fe}_3\text{C})_n$  is the only product from annealed steels, would be very strong evidence that the  $(\text{Fe}_2\text{C})_n$  is precipitated or crystallized as such from solid solution during slow cooling and is not derived as a decomposition product of  $(\text{Fe}_3\text{C})_n$ . Whether  $(\text{Fe}_2\text{C})_n$  is a constituent of the pearlite or of the free cementite in slowly cooled white iron, we are at present unable to say.

A mixture of carbides  $(\text{Fe}_3\text{C})_n$  and  $(\text{Fe}_2\text{C})_n$  to contain 8.20 per cent carbon and 91.80 per cent iron, would have to be made up of 49 per cent of the former and 51 per cent of the latter carbide. If all the carbon and iron recovered as carbide described in Table I represents a mixture of the two carbides recovered from 302.05 grams of metal, there would be 17.03 per cent of  $(\text{Fe}_3\text{C})_n$  and 17.73 per cent of  $(\text{Fe}_2\text{C})_n$ , or in all 34.76 per cent of mixed carbides derived from the electrolytic treatment of slowly cooled white iron.

When pieces of the same white iron employed in the above-described work were electrolyzed without first annealing the iron, samples of carbide were recovered containing in many cases much more carbon than was found in the carbides recovered from the unannealed metal. In the case of the carbides recovered from the unannealed metal, the sum of the iron and carbon was far short of 100 per cent, and the difference increased as the percentage of carbon in the residues increased, thus showing that the large amount of carbon was due to decomposition.

It was thought that possibly a microscopic examination of



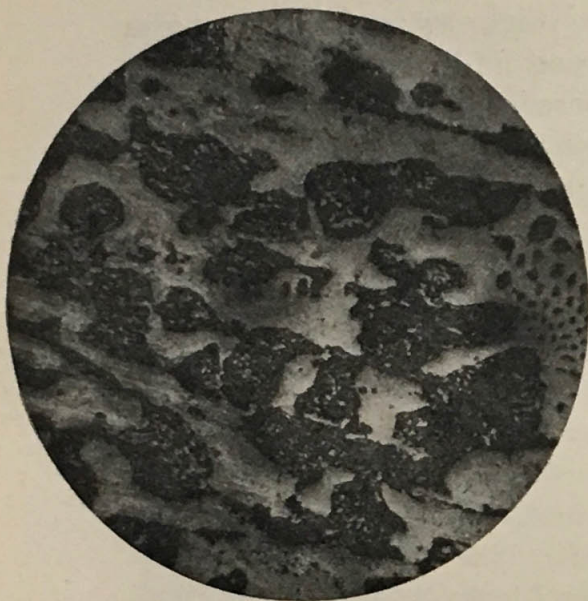


Fig. 1. White charcoal Pig Iron, unheated.  
Magnified 110 diameters.

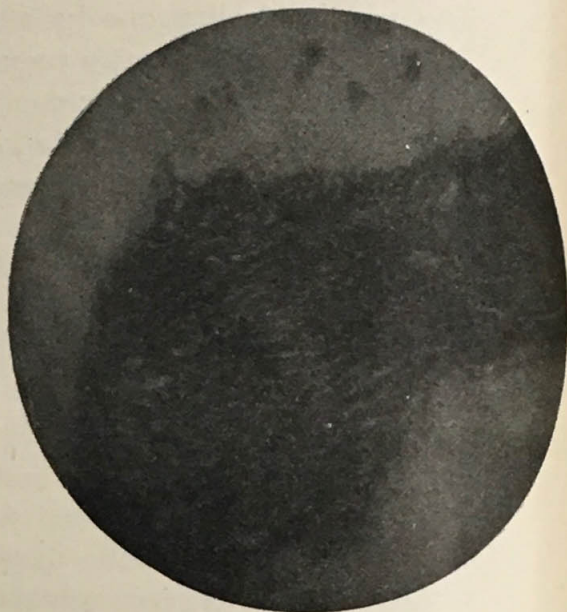


Fig. 2. Same as Fig. 1.  
Magnified 480 diameters.

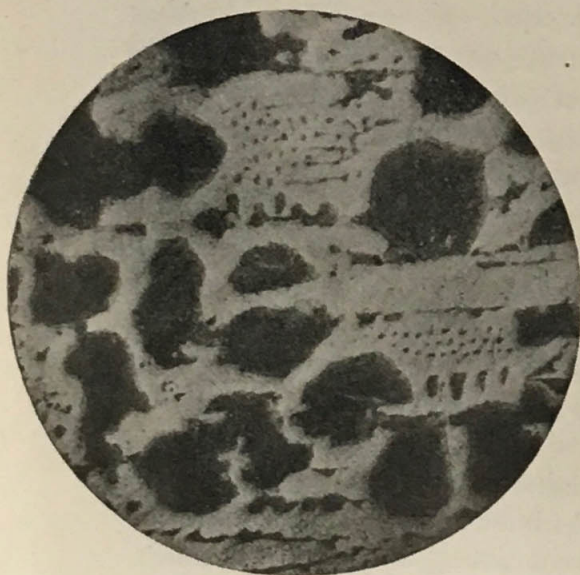


Fig. 3. Same Pig Iron reheated to 950° C.  
and slowly cooled.  
Magnified 110 diameters.

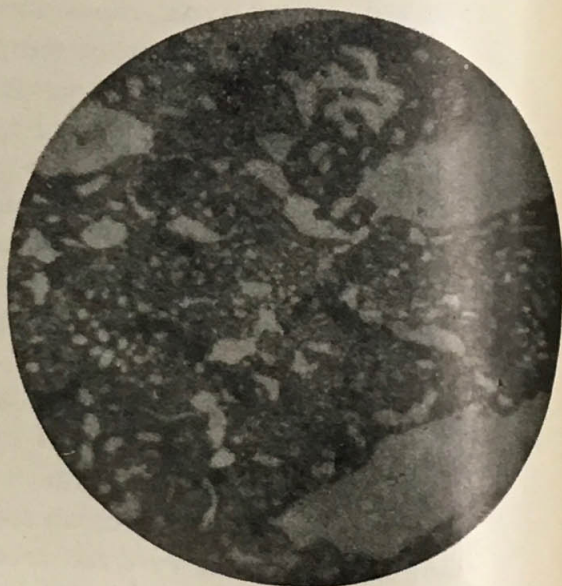


Fig. 4. Same as fig. 3.  
Magnified 480 diameters.

the white iron, before and after annealing, might throw some additional light on the change which took place during the slow cooling. No positive information, however, seems to be obtained

by this method of examination. I am indebted to Professor Albert Sauveur, who has kindly offered to make the metallographic examination of the white iron, before and after annealing. His report is as follows:

#### DESCRIPTION OF MICRO-STRUCTURE

*Sample I.* — White charcoal pig iron, untreated.

The structure of this sample is illustrated in photo-micrographs Nos. 1 and 2, magnified respectively 110 and 480 diameters. They reveal the normal appearance of white cast iron, the structure being made up of a matrix of cementite and numerous globular lumps of pearlite.

In Fig. 2 is shown the internal structure of one of the pearlite grains. It presents the laminated structure characteristic of normal pearlite and of the majority of eutectic alloys.

*Sample II.* — Same pig iron re-heated to  $950^{\circ}$  C., and slowly cooled.

The micro-structure of this sample is illustrated by photo-micrographs 3 and 4, under magnification respectively of 110 and 480 diameters. It will be seen that the treatment to which this sample was subjected has not altered the general appearance of the structure, i.e., the relative proportion of cementite and pearlite, and the size and distribution of the pearlite areas. It has, however, greatly modified the internal structure of the pearlite particles. The pearlite no longer exhibits the characteristic lamination noted in the untreated sample. It is now made up of irregular particles of a white constituent (cementite?) embedded in a dark matrix (ferrite?).



## NOTE ON THE INFLUENCE OF THE RATE OF COOLING ON THE STRUCTURE OF STEEL\*

By ALBERT SAUVEUR and H. C. BOYNTON, Cambridge, Mass.

IN the course of some experiments conducted in the Metallographical Laboratory of Harvard University, some interesting facts were brought to light which appear to be worth recording in advance of a more elaborate and exhaustive paper which the authors hope to present to the Institute in the near future.

Fig. 1 shows, under a magnification of 100 diameters, the microstructure of the cross-section of a steel bar  $\frac{1}{2}$  inch square, containing 0.52 per cent carbon, heated to  $1,100^{\circ}$  C. and cooled slowly with the furnace. Fig. 2 illustrates the structure of the same steel, heated to the same temperature but cooled more rapidly in the air.

Both samples were cut from the same bar and heated side by side in the furnace. When a temperature of  $1,100^{\circ}$  C. had been reached, one sample was taken out of the furnace and allowed to cool in the air, while the other was cooled with the furnace. The only difference, therefore, in the treatment of both samples will be found in their respective rate of cooling. This variation in the rate of cooling resulted, as shown here, in a very marked difference in the structure of the two samples.

The air-cooled sample has assumed a sharply defined network structure, while the furnace-cooled specimen possesses a structure which, for lack of a better term, we shall call a granulated structure.

Similar experiments were repeated a great many times with identical results. Air-cooling from temperatures exceeding  $900^{\circ}$  C. always resulted in the formation of a network structure, while very slow cooling in the furnace, from similar temperatures, was always accompanied by the formation of a granulated structure.

The examination of these two structures cannot fail to impress us with the important part played by the rate of cooling from a high temperature upon the constitution, and, therefore, upon the properties of medium hard steel, and probably of all steels.

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\* Paper presented to the American Institute of Mining, Albany Meeting February, 1903.

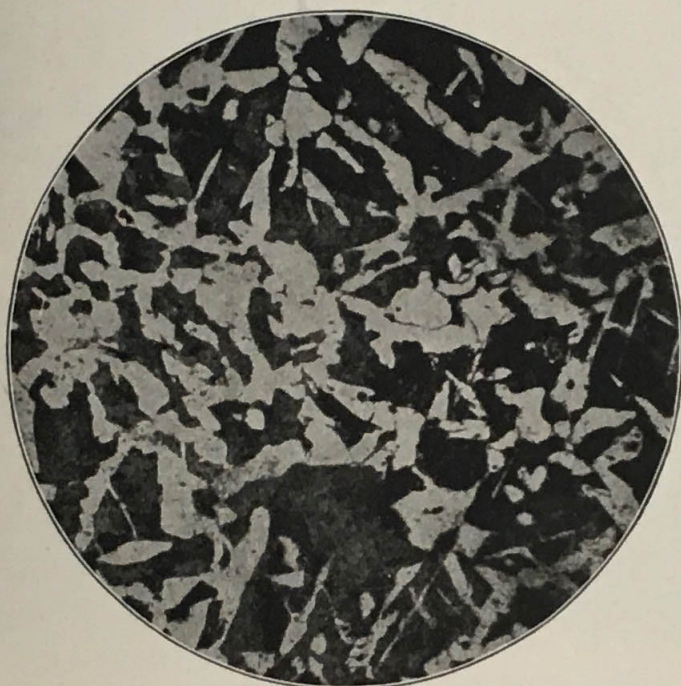


Fig. 1. Steel bar containing 0.52 per cent carbon, heated to  $1100^{\circ}\text{C}$ . and cooled slowly with the furnace. Magnified 100 diameters.



Fig. 2. Same steel heated to  $1100^{\circ}\text{C}$ . and cooled in air.



More thoughtful consideration will lead to other conclusions and speculations which are not without scientific as well as practical interest.

It is a well-acquired fact that all the carbon present in undersaturated or hypo-eutectic steel is included in the constituent pearlite. It is also generally stated that pearlite has a constant composition, its carbon content being in the neighborhood of 0.8 per cent. Finally, undersaturated steels, when unhardened, are always described as being made up of ferrite and pearlite, which implies the assumption that the dark constituent of these steels is always made up of pearlite. Upon examination of Figs. 1 and 2, however, it will be found that the dark constituent occupies a much larger proportion of the mass in the case of the air-cooled sample. A rough estimate will show that the dark constituent occupies a little more than one-half of the area of Fig. 1, while it occupies at least 90 per cent of the area of Fig. 2. It is evident, therefore, that the carbon in Fig. 1 has segregated into a smaller bulk, or, in other words, the dark constituent of the furnace-cooled sample contains a larger proportion of carbon than the dark constituent of the air-cooled sample. If we call the dark constituent of both samples pearlite, then we must infer that the composition of pearlite, instead of being constant, varies with the rate of cooling. Such conclusion, however, would be so much opposed to many important evidences which we have concerning the constitution of pearlite as to be quite untenable.

To throw more light upon the nature of the dark constituent of Figs. 1 and 2, the same samples were examined and photographed under higher magnification, as shown in Figs. 3 and 4. The high-power photograph of the furnace-cooled sample includes a particle of ferrite and some of the surrounding dark constituent. It will be noticed that the dark constituent exhibits everywhere the characteristic structure of pearlite, which is also that of nearly all eutectic alloys: It is made up of parallel plates alternately of one and the other constituent (of ferrite and cementite in the case of steel). In Fig. 4 is shown a high-power photograph of one of the meshes of the network of the air-cooled sample. The dark constituent is only partially laminated; in many places its structure is ill-defined; it does not possess, to the same degree, the structural characteristics of eutectic alloys.

The inference from these considerations must necessarily be,

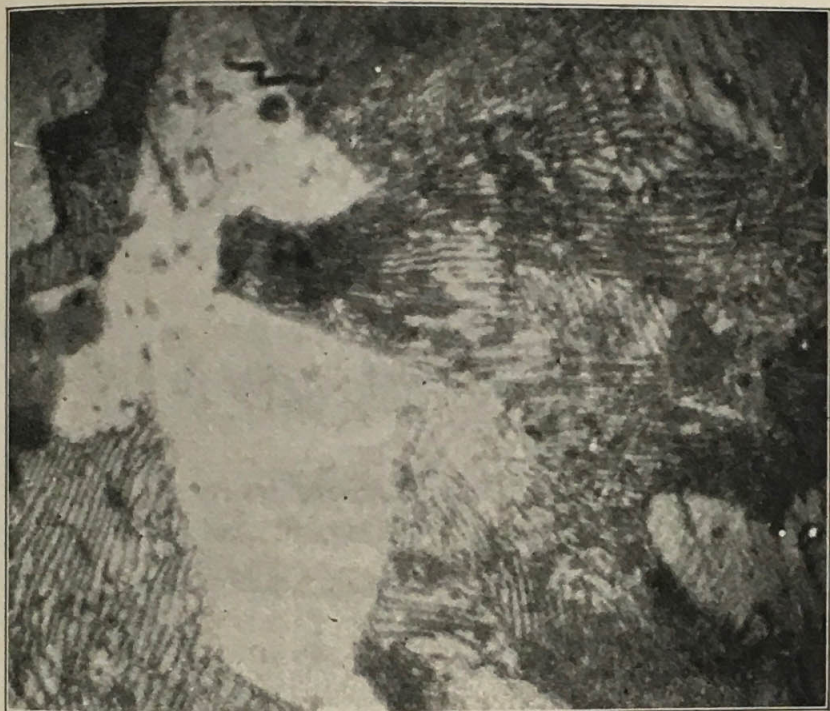


Fig. 3. Same as Fig. 1. Magnified 1000 diameters.



Fig. 4. Same as Fig. 2. Magnified 1000 diameters.



that while the dark constituent of the furnace-cooled sample is true pearlite, the corresponding constituent in the air-cooled sample is not true pearlite; it contains more iron (more ferrite) than true pearlite, evidently because the time necessary for this excess of ferrite to segregate (to be expelled) was denied by the relatively rapid cooling of this sample.

The mechanism controlling the formation of the structure is apparently as follows: At a high temperature, the whole of the carbon is diffused through, or dissolved in, the iron. Steel at this temperature has rightly been compared to a solid solution of iron and carbon. This solid solution can be retained in the cold (partially, at least) by sudden cooling, the resulting constituent being then called martensite. Upon slow cooling, however, the solution is unable to retain the whole of the iron; as the metal cools, some ferrite falls out of solution, and if the cooling be sufficiently slow a stage of equilibrium is reached at each temperature. When the critical temperature is attained, the remaining solution of iron and carbon reaches the composition of the eutectic alloy (that is, of pearlite), and in passing through the critical range the carbon enters into combination with some of the iron still holding it in solution, to form the carbide  $\text{Fe}_3\text{C}$ , or cementite. If the cooling is not sufficiently slow, however, to allow this stage of equilibrium to be reached, some of the iron is retained by the dark constituent, which therefore never reaches the composition of true pearlite, and which might then be called dilute pearlite or ferrous pearlite.

It is evident, from what precedes, that in order to induce the formation of true pearlite in steel, the metal must be cooled sufficiently slowly from a temperature exceeding, say,  $900^\circ \text{C}$ ., to, and past, the critical point; the rate of cooling below the critical point being quite immaterial. If the time necessary for the formation of true pearlite be denied, then the dark constituent present in steel is of a different character: it contains a smaller proportion of carbon, and assumes, imperfectly and locally only, the structural characteristics of pearlite.

It should also be remembered that since the formation of true pearlite depends upon a sufficiently slow rate of cooling, this constituent will be more readily formed in large pieces than in those of small dimensions.

The authors are well aware that other writers have alluded



to this character of the dark constituent of relatively quickly cooled steel, but they trust that they have brought these facts to the attention of those interested in steel more forcibly than has ever been done before.

The dilute or ferrous pearlite just alluded to, and resulting from the relatively quick cooling of steel from a high temperature, recalls the constituent which Osmond has called sorbite, the existence of which has been and is still doubted by some metallographists. Sorbite, however, results essentially from a relatively quick cooling *while the metal is passing through the critical range*, in such a way that the critical transformation which results in the production of true pearlite is not fully completed. The constituents being considered here, on the other hand, is the result of relatively quick cooling *above the critical point*, so that it retains more ferrite than true pearlite, and occupies a larger proportion of the mass. Osmond's sorbite does not necessarily contain more ferrite than true pearlite. Looking into the matter more closely, however, it will be seen that a relatively slow cooling to the critical point almost necessarily means the same rate of cooling through the critical point; so that the characteristics of sorbite will also be developed in this dilute pearlite, unless the cooling be conducted in a very peculiar way, consisting of rapid cooling to the critical point, and of slow cooling through that point. It appears to the writers that the dilute pearlite with which they are now concerned may properly be called sorbite. To select another name for it might lead to confusion.

Most forged steel implements are air-cooled, which means, in the majority of cases, too rapid cooling for the production of true pearlite. The dark constituent of forged steel which is always referred to as pearlite is seldom true pearlite; in the majority of cases it is sorbite. Witness the network structure so frequently found in steel rails containing some 0.50 per cent carbon, and which is so similar to the structure shown in Fig. 2.

It was long ago proposed to divide pearlite into two varieties, the lamellar and the granular. It appears, however, that if the dark constituent does not exhibit the laminated structure of pearlite, it consists of sorbite, and not of true pearlite. Granular pearlite is not pearlite at all, but sorbite.

It is evident, moreover, that the carbon content of sorbite will vary with the rate of cooling. It is, as Osmond has rightly



said, a transition constituent, marking a step in the transformation of martensite into pearlite. The quicker the cooling the more it will approach the nature of martensite, and *vice versa*.

Several interesting questions are suggested by this varying composition of the dark constituent of unhardened steel. Some of them will be briefly mentioned here.

1. What is the difference between the properties of a steel whose dark constituent consists of true pearlite, and those of the same metal whose dark constituent is made up of sorbite?

Samples of steel containing 0.55 per cent of carbon, and to which the two kinds of structure had been imparted, were tested, and it was found that the network-structure — that is. the structure in which the dark constituent consists of sorbite — corresponded to a greater elastic limit, and greater tenacity, but to a smaller ductility. Some of the results are given below:

Treatment of Sample	Character of Dark Constituent	Elastic Limit. Lbs per Sq. inch	Tensile Strength. Lbs per Sq. inch	Elongation. Per Cent in 8 inches	Reduction of Area. Per Cent
Heated to 1150° C. and cooled with furnace . .	Pearlite	39,901	81,162	17	27.61
Heated to 1150° C. and cooled in air . . . . .	Sorbite	53,260	99,979	12	20.55

2. What are the properties of pearlite compared to those of sorbite? To answer this question 2 samples of steel, in the shape of bars  $\frac{1}{2}$  inch square, should be procured, containing respectively about 0.60 and 0.80 per cent of carbon, and as far as possible containing the same percentages of other impurities. The first sample should be heated to, say, 1,000°, and cooled in the air. We should expect to find that it consists then of a mass of sorbite, as time had been denied for the excess of iron to segregate as ferrite. The second sample should be heated to the same temperature but cooled slowly in the furnace. It will then consist of a mass of true pearlite.

The physical testing of these two samples will indicate the respective properties of pearlite and sorbite (at least of the sorbite formed under the conditions just indicated).

3. Seeing that sorbite is a transition form between martensite and pearlite, does it not also contain its carbon in a transitional form between hardening carbon (the condition of carbon in martensite) and cement carbon (the condition of carbon in pearlite), or partly as hardening and partly as combined carbon? Careful analysis by the colorimetric method of samples of the same steel, but containing respectively sorbite and pearlite, should throw much light upon this point. It can be reasonably expected that the analysis of the pearlite steel will give higher results than those of the sorbite steel.

4. When it is attempted to judge of the amount of carbon present in a sample of steel by the appearance of its microstructure, the variation of the area occupied by the dark constituent, with the rate of cooling, as described in these pages, should be carefully borne in mind. otherwise serious errors will be made. It would appear that, in order to apply this method with any degree of accuracy, it is necessary first to treat the samples so as to cause the formation of true pearlite. With this precaution, the authors believe that the method will give results comparing favorably in accuracy with the colorimetric methods.

These points and a number of others are being investigated in the metallographical laboratory of Harvard University, and the authors hope to be able, before long, to publish some of their results.

## ON CERTAIN PROPERTIES OF THE ALLOYS OF THE GOLD-SILVER SERIES \*

By the late Sir W. C. ROBERTS-AUSTEN and T. K. ROSE

IN a former communication to the Society† the curve of the initial freezing points of the alloys of gold and copper and some micrographic evidence as to their structure were given, and it was shown that according to the theory of solutions the alloys rich in gold should not be homogeneous after they have solidified. The fact that they are not uniform was confirmed by analysis. The subject has, however, more than theoretical interest, and the

\* Read before the Royal Society, December 11, 1902.

† *Roy. Soc. Pro.*, Vol. 67, 1900, p. 105.



inference was drawn that standard gold, which consists of eleven parts by weight of gold to one of copper, is unsuitable as a material for the preparation of the trial plates by which the standard of the coinage is tested. These trial plates according to law must contain 916.6 parts of gold and 83.3 of "alloy," that is of some other metal, and it remained to be determined what the other metal should be.

It will be at once apparent that the alloy or mixture of the two metals must, if the cold mass is to be uniform, solidify as a whole, that is to say, that the crystals first formed should be of

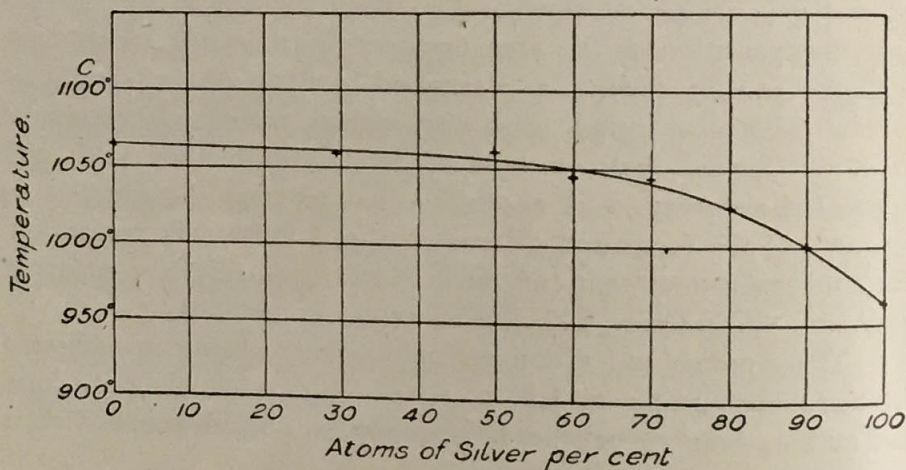


Fig. 1.

the same composition as the mother liquor, and this condition can be fulfilled by isomorphous mixtures only. It has long been recognized that the gold-silver alloys are cases of isomorphism, and Gautier, in 1896, stated\* that the freezing-point curve of the series followed a straight line if the percentages by weight of the constituents were taken as abscissæ.

This curve was re-determined by experiment, a number of alloys being made up and autographic records taken of their cooling curves by the Roberts-Austen recording pyrometer. The results obtained are given in the following table, and have been plotted in Fig. 1, in which the abscissæ are atomic proportions of the metals in the alloys. The freezing point of gold was taken as 1,064°.

\* *Bull. de la Soc. d'Encouragement*, Oct., 1896.

## Percentage of gold present in alloy

By weight	In atoms	Freezing point
100	100	1,064°
80.99	70.25	1,061
64.60	49.97	1,061
54.80	39.89	1,046
43.98	30.07	1,044
31.71	20.28	1,028
17.23	10.23	1,001

The following points had been observed by Heycock and Neville:\*

2.26	1.25	962°
0.91	0.50	961
0	0	960

It will be seen that Gautier's conclusion is substantially confirmed, but it was observed, as one of us had previously pointed out,† that the first additions of silver did not depress the freezing point of gold. So far does this property extend that even the alloy containing 50 atoms of gold to 50 of silver, or 64.6 per cent of gold by weight, solidifies at 1,061°, which is only 3° below the freezing point of pure gold. With further additions of silver there is a steady acceleration in the rate of lowering of the point of solidification so that the freezing-point curve of the series has no double flexure, unless one is indicated near the silver end of the curve by Heycock and Neville's results.

There is, of course, no eutectic alloy observable in any member of the series.

The alloys all consist of large grains, but these are built up of smaller grains so that the ultimate structure is exceedingly minute. When magnified 1,500 diameters the grains appear as small irregular crystals of the cubic system (see Fig. 2). In order to develop any segregation that might take place, an ingot of the standard alloy containing 91.66 per cent of gold by weight was heated for 2 months in one of the annealing furnaces at the Royal Mint, the temperature of which was kept at about 700° by day, but fell to about 100° at night. The maximum temperature

\* *Phil. Trans.*, A, Vol. 189, 1897, p. 69.

† Roberts-Austen, *Proc. Inst. Mechanical Engineers*, 1891, p. 564 (14769).



attained was over  $300^{\circ}$  below the fusing point of the alloy, and the sharpness of the angles of the specimen had suffered no change. After this treatment it was found that the grains had increased in size, and the crystals forming them had become well developed, as shown in Fig. 3, in which the structure is magnified 1,500 diameters. No true segregation, however, could be detected even in this ingot, either by analysis or by the microscope, and plates prepared by rolling out ingots containing 916.6 parts by weight of gold, and 83.3 parts of silver, were found on analysis to be uniform in composition.

The ancient trial plates, according to the analysis made by

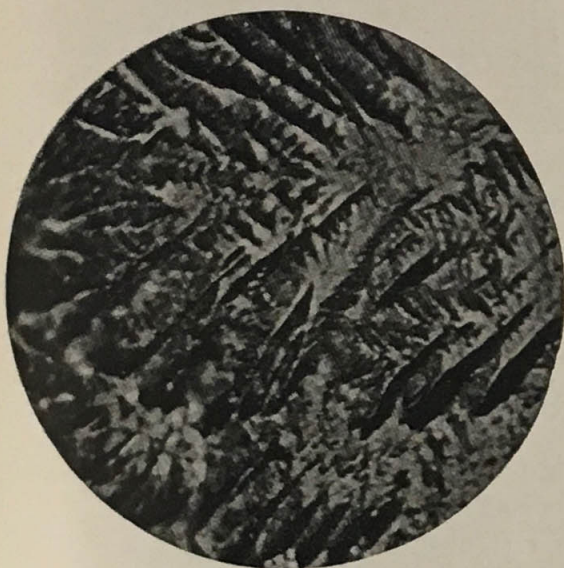


Fig. 2.

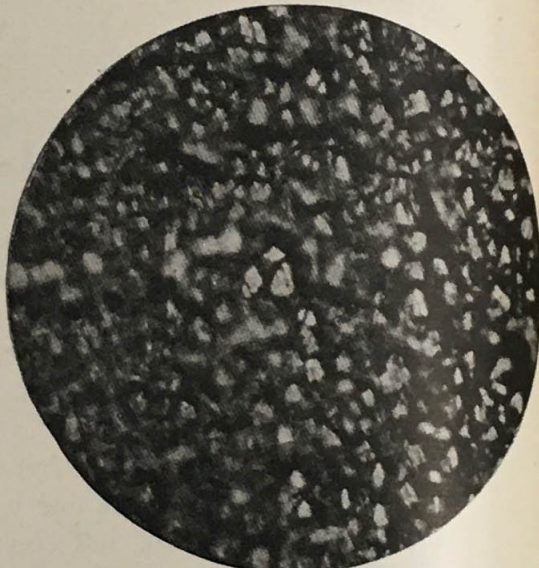


Fig. 3.

one of us,\* consist of a triple alloy of gold, silver, and copper. The earliest one in existence was made in 1527, the year following the first introduction of the standard 916.6. This plate contained only 0.62 per cent of copper, and was probably intended to consist of gold and silver only. All subsequent plates, however, down to that made in 1829, contained much larger amounts of copper. In 1873 it was determined to omit the silver and to use only copper as the alloying metal, and thus to preserve identity of composition between the trial plate made in that year and the

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\* Roberts-Austen, *Chem. Soc. Journal*, 1874, p. 197.

coinage. In view, however, of the importance of obtaining homogeneous trial plates and of the ease with which the exact quantity of copper required to make the assay pieces identical in composition can be added to the pieces of the trial plate during the course of the assays, it is preferable to use only silver as the alloying metal in the manufacture of the trial plates.

Such an alloy has accordingly been used at the Royal Mint since the beginning of the present year instead of fine gold for checks in the assay of standard bars and coins. In view of the minute accuracy with which the operations of coinage have to be conducted, this is a matter of much importance. By this method any errors are avoided which might be caused by accidental variations in weights occurring after the trial plates have been made.

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### CONCERNING THE RAIL SITUATION \*

THE special report made by Mr. Dudley to Mr. Wilgus, which is printed on another page, gives an opportunity to say a word about the rail situation. It will be remembered that the American Society of Civil Engineers has a special committee (appointed a few months ago) to consider the whole subject of the rail sections, of mill practice and of specification and inspection. Quite lately a committee of representatives of the mills has been formed to work with the committee of the American Society of Civil Engineers. Last week these two committees met in New York. separately at first, and then in joint session. It would not be appropriate or desirable to report in detail the discussions at those sessions, even if we knew them. They were quite informal. The main object, as we understand, was to clear the ground and to prepare for common action.

It may not be amiss to state again the main facts of the situation.

First: Probably everybody who has paid any attention to the metallurgy of steel in recent years, and particularly to making rails, is by this time thoroughly aware of the fact that the

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\* *The Railroad Gazette*, January 30, 1903.



structure of steel depends much upon the finishing temperature; and in consequence of the spread of this knowledge there has come about a definite and general notion that the final passes on rails must be made at a lower temperature than is now common. Nobody disputes this position.

Second: It is pretty generally conceded that the railroads are disappointed in the results which they have got from the recent heavy sections. They do not get service enough to compensate for the money paid for the additional weight. This is matter of general knowledge. A chief engineer has lately said to us. "The combination among the rail makers is resulting in a constant deterioration in the quality of the material we are receiving and we ought to do all we can to counteract this tendency." We do not accept or reject this opinion; we merely give it as a specimen of what we often hear.

Third: It has been thought by a good many people that a still better balance of the standard sections would help to secure a lower finishing temperature and, consequently, a better internal structure. This means a transfer of a part of the metal put in the head of the American Society standard sections to the flange, and this idea probably had more to do than any other one consideration in bringing about the appointment of the American Society's committee; although it is only one of the considerations which led to the formation of that committee.

Fourth: It has been maintained and is maintained by many that a shrinkage clause in rail specifications would help to secure a lower finishing temperature. That is, it is obvious that if, for example, saws for a 30-ft. rail are set 30 ft. 7 in. apart the rail can be delivered to the saws hotter than if the saws are set 30 ft.  $5\frac{7}{8}$  in. apart. There are makers who do not believe that a shrinkage specification will control the structure of the steel even if it does control the finishing temperature.

Fifth: Various methods of mill treatment are now used to secure the results which everybody knows can be secured by thorough working at low temperature. The Kennedy-Morrison process, as used at the Edgar Thomson mills, is one of these. That process delays the rails before they reach the finishing rolls, and must be familiar to our readers. The method which is practiced at Sparrows Point is described in Mr. Dudley's report printed in this issue.



This is a short statement of the situation. The American Society's committee, so far as we are informed, would not now recommend any change of section. What it may be led to do by further information of course we shall not try to conjecture. The committee would not with its present information try to lay down specifications of chemical composition or mill treatment. The attitude of the committee, so far as we can judge, is to let the makers agree as to what is necessary in order that they may give the results sought, namely, a better and more constant quality of finished steel. If the rail makers agree that it will be for the interest of the art to make some modification in the American Society's standard sections, we suppose the committee will recommend such modification. The representatives of the rail makers, so far as we are informed, do not think that any change of section is necessary, but on this they are not agreed, and this appears to be only a tentative opinion.

As the matter now stands, the American Society's committee will probably state with some definiteness its questions and requirements and the rail makers' committee will take these up and answer them as from the whole body of manufacturers. This, we believe, is a fair statement of the present situation, and this situation seems very promising. The attitude of the engineers is conservative and reasonable, and the attitude of the rail makers is reasonable and liberal, and it is pretty certain that good and lasting results will be worked out by the two committees. Obviously, a matter so intricate, involving so many disputed technical points, to say nothing of business interests, cannot be settled at once out of hand.



## NON-EXPANSIVE ALLOYS\*

By C. E. GUILLAUME

THE criticism by Dr. Stillman, at page 1075, Vol. 25, of the new nickel steel has not surprised me in the least. It is certainly difficult to imagine, *a priori*, how it can be that the mixture of iron and nickel, of which the expansions are respectively 11.5 and 12.5 millimeters, can give a substance having sensibly no expansion. Yet the fact exists, and alloys with little expansibility exist to-day — prepared no longer as curiosities of the laboratory, but in the state of industrial products, of which the employment increases from day to day, as I will indicate later on.

Assuredly it is not very easy to understand how — that is to say, by the action of what secret mechanism — the alloys, sensibly non-expansive may exist. For several years I have tried to show an outline of their theory, founded on the idea of a chemical equilibrium variable with the temperature, and it is to this general idea that the discussion pertains still; but under this vague form, the theory is not sufficiently explicit, and I should admit that all the attempts at specialization still encounter some difficulties. Yet one will readily recognize the idea of M. Osmond, according to which the passage of iron from the state *Gamma* to the state *Alpha* by lowering of the temperature ought to play an important rôle in the phenomena in question. It suffices, in effect, to admit that this passage, which is produced in isolated iron, at an elevated temperature, and in a limited interval, is brought down to ordinary temperatures and spread over a great interval, in order to explain perfectly the existence of slightly expansible alloys. This theory is supported by a great number of facts, but, looking closely, one will be convinced that it is yet incomplete. Meanwhile it will be provisorily admitted as one of the most probable particularizations from the general idea that I have put forth.

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\* Being a letter sent to the *American Machinist*, Jan. 8, 1903, by Mr. C. E. Guillaume, the distinguished associate director of the International Bureau of Weights and Measures, whose fruitful experiments into the properties of Nickel-Iron Alloys have been so skilfully conducted.



The publications that I send you will give you an idea of the various properties of the new alloys. You will see further that this has no longer the aspect of an isolated observation, early given publicity, but indeed a series of investigations systematically conducted through the coöperation of that powerful metallurgical organization, the Société de Commentry-Fourchambault at Decazeville, with the International Bureau of Weights and Measures, specially equipped for this sort of research.

Not only the conditions of the existence of slightly expansible alloys have been fixed, but further, this existence has been connected with a series of anomalies which embrace all the alloys of iron and nickel and extend to all their properties.

Few publications on these questions have hitherto been made in the English language; yet a demonstration of the properties of these alloys was given in the Royal Society Soirée of May 19, 1897, to which I was graciously invited. It was repeated by Sir W. Roberts-Austen, at the meeting of the Britannic Association, which took place at Toronto, in the autumn of the same year. Finally, an article appeared in the *Engineering Magazine* of October, 1901.

The applications of nickel steels founded on their anomaly of expansion have not delayed in coming forward. Today there is not a clock of precision manufactured in Germany that does not carry a pendulum with a nickel-steel rod, allowing the disuse of mercury for compensation. If I cite particularly Germany for this application, it is because the new pendulum has spread there with astonishing rapidity; but they are made in other countries also; notably France and Switzerland, although less exclusively.

Among the scientific applications of slightly expansible alloys, I will cite in the first place their employment in geodetic instruments whether for the measurement of bases, or even in theodolites for the measurement of angles, as well as in the leveling apparatus adopted by the United States Coast and Geodetic Survey. Base instruments have already been delivered or are under construction for the geodetic services of Germany, France, Japan, Mexico, Roumania and Russia. Others have been employed by the Swiss-Russian expedition of Spitzbergen, by Sir D. Gill in Cape Colony and Rhodesia, by the geographical service of the French army, and by the hydrographic service



of the navy at Madagascar, at Tonkin and in other colonies. And especially the very small expansion of the instruments has rendered the service the greater as the measure of the temperature was the more difficult.

Another anomaly intimately connected with that to which I have alluded has allowed another application quite unexpected. It is known, thanks to the work of Dent, that a chronometer compensating by ordinary means for two temperatures does not do so for others. In recent decades the horologists, especially in England, have put forth much ingenuity and great deftness to obtain, by mechanical processes, a complete compensation. They have practically accomplished the task in many cases, but with an appreciable increase of complication in the construction of the chronometers.

But by suitably combining in a balance of ordinary form a nickel steel properly chosen, with the brass generally employed in the "*belame*," a complete compensation is effected while preserving a stability of movement which leaves nothing to be desired. On this question, the last report of the Director of the Observatory of Neuchâtel is very explicit.

Finally, it may be remarked that, by adding iron or nickel to the least expansive alloy, alloys may be obtained possessing such expansion as is desired. The most important, from an industrial point of view, is certainly that which possesses the expansion of glass, which hitherto has only been reproduced among the metals by platinum. Owing to the unstinted consumption of platinum, which is needed in the manufacture of incandescent lamps, the price has become exorbitant, which renders it less and less available within the means of laboratories.

For some time the Société de Commentry-Fourchambault et Decazeville has been delivering commercially under the name *platinite* an alloy which absolutely replaces platinum for this particular use and several important lamp factories have completely renounced the employment of the precious metal. Up to the present, several tens of kilogrammes of platinum have thus been rendered able to return to circulation, and I do not doubt that when this application of the new alloys becomes better known, almost all the platinum used in lamp manufacture, and which forms nearly a third of the annual production, will be returned to the industries which cannot dispense with it and to the lab-

oratories which, by reason of the high price of the metal, are obliged to use less and less.

## THE MICROSCOPE IN CRUCIBLE STEEL MANUFACTURE\*

By JAMES J. MAHON

THE technical journals in the past few years have devoted considerable space to the microscope and its relative value in the manufacture of fine steel. Practically all the experiments made and illustrations shown indicate that the microscopic examinations have been made after the steel has been subjected to some form of treatment. The writer's opinion is that the microscope, in order to be of any practical assistance to the manufacturer of fine steel, should be used on the ingot immediately after it is cast. The manufacturer of good crucible steel must in the first place use the best of materials, principally good iron. Good steel cannot be made from poor iron, even with the assistance of the microscope. As soon as the ingot is cast, it is then either good steel or bad steel, and its qualities should be determined right then and there. If the ingot shows that the refining process has been incomplete and consequently the elements not properly combined, there is only one treatment which the ingot should undergo and that is to cut it up and melt it over again. While we hear and read a great deal of the results of different forms of heat treatment, we must still bear in mind that while good steel can be spoiled by bad treatment, bad steel cannot be converted into good steel by any kind of treatment except by remelting it. Consequently the time to determine the quality of the steel is immediately after it is cast. The microscope has demonstrated one fact, and that is that it is impossible to determine whether an ingot has been properly melted by an examination of the fracture with the naked eye. The most essential thing in connection with the manufacture of high-grade crucible steel is the amount and quality of iron which the mixture contains. The quality of the iron cannot be determined by chemical analysis alone, as some makes of iron,

\* *The Iron Age*, Jan. 8, 1903.



although very low in both phosphorus and sulphur, do not make as fine a quality of steel as other makes of iron which contain double the amount of those impurities. Although at the present time there are many ways of determining the qualities of iron for melting purposes, at the same time it is the writer's opinion that the microscope can be of considerable assistance to the manufacturer in selecting iron which will have the capacity to withstand the strains and work put upon it after it has been converted into steel. The next essential thing is to properly refine or melt the charge which the crucible contains. The temperature required to do this depends upon what the mixture is composed of. The old process of "killing" is all right so far as it goes, but what is more essential than the "killing" or the length of time it takes to perform the operation is the temperature of the metal during the operation. In fact, it is possible that the mixture may be composed of such materials that they cannot be combined into a homogeneous state in a single operation. The next question to consider after the ingot is cast is whether the refining process has been complete, whether the ingot is practically homogeneous or not. Until that point can be determined, why go any further and waste time and money without getting any positive results? Especially is this so when conducting costly experiments with steel made from different combinations. It is not much satisfaction to find out with the assistance of the microscope after the tests are all over that some of the steel had not been properly melted. The microscope can be of great assistance to the steel manufacturer, not as an instrument to be used at the post-mortem examination to determine the cause of death of the patient, but rather as an instrument to detect the source of the ailment while the patient is still in a curable condition.\*

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\* While we do not desire to deny the value of the microscope when applied in the way suggested by Mr. Mahon, we believe that he greatly underestimates the assistance to be derived from post-mortem examination. If the result of such examination is to prevent the death of other patients from the same disease, then the importance of the method is demonstrated and this, indeed, is precisely what it does. The necessary death of a few to save many is of relatively little moment. Special subjects for post-mortem examination, moreover, may be prepared, whose loss need not be mourned by any one. This method which has already thrown so much light upon the rational treatment of iron and steel will, we believe, continue to be the most fruitful.—*Ed.*

## EUTECTIC OR BENMUTIC? \*

M. Osmond uses the expression "hyper-eutectic steel" to indicate steel containing more than the pearlite ratio of 0.80 per cent of carbon, and "hypoeutectic steel" to indicate steel containing less than that. In the same way he would no doubt speak of eutectic steel as steel containing exactly this pearlite ratio. This last, however, brings up a serious objection to the use of these expressions.

The eutectic, following Guthrie's original use of the word, is the most fusible alloy in a given series of alloys, or in a certain range of that series, and eutectic steel should therefore mean the most fusible steel. M. Osmond applies the term to steel of the pearlite ratio, not because that is the most fusible steel, but because it is the steel of the lowest transformation point, i.e., he borrows this term "eutectic," which clearly referred to solidification, and applies it to phenomena of transformation within the already solidified mass. This is a very natural and at first sight proper course, but the serious objection to it is that steel of this composition is not eutectic in Guthrie's sense because it is not the most fusible steel. M. Osmond applies the term to steel of the actually found much confusion to arise from this use of the term with students, and as a teacher I write this note to suggest a different term.

It appears to me that it would be well to indicate the fact that this steel is the steel of lowest transformation point as distinguished from lowest melting point, by using an adjective which refers distinctly to transformation points and does not refer to melting points, for it is by neglect of this that M. Osmond's expression "eutectic steel" leads to confusion. What we need, then, is some term which shall clearly indicate that the steel is of the lowest transformation point, and the next question is, what term shall we use?

We naturally turn to the Greek, but unfortunately there is no short Greek word to represent transformation. Eutectic means "well melting," and the corresponding term to express steel

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\* This article appeared, as a letter to the Editor, in the *Engineering and Mining Journal* for January 24, 1903, and the discussion which follow was also addressed to this Journal and published respectively in the issues for February 28, March 7 and 21.



or other alloys of lowest transformation point might be either "eumetallactic" or "eumetamorphic."

Both of these words seem to be wholly unsuitable. "Eumetallactic" means literally "well changing," but then everybody would suppose the letters "m-e-t-a-l" referred in some way to the common English word "metal," and their reference to transformation would not be seen. "Eumetamorphic," on the other hand, is intolerably long and cumbersome. If we were to speak of "hyper-eumetamorphic steel" we should use shocking jargon.

Under these conditions, I propose that we turn to Latin, and I suggest the word "benmutic," which evidently means "well-transforming," and therefore is quite parallel with Guthrie's term, "eutectic" or "well melting." The expression "benmutic steel" or "benmutic alloy" would mean the steel or alloy of the lowest transformation point, and it would not confuse the reader by needless reference to the melting point. In this case "benmutic steel" would be that with 0.80 per cent of carbon. Steel with less than this amount would be called "sub-benmutic" and that with more than this amount "super-benmutic." I can see no objection to the expressions, but I should be glad to hear how they strike your readers.

If this term was adopted, then the term "eutectic iron" should be held to refer strictly to iron containing 4.3 per cent of carbon, i.e., the iron of the lowest melting point and "hypoeutectic iron" and "hypereutectic iron" should respectively refer the former to iron containing less than 4.3 per cent of carbon, and the latter to iron containing more than this amount.

There is another point in which the nomenclature of metallography seems to need an addition. Above the critical range,  $A_1$  to  $A_3$ , the iron and carbon of steel unite to form a solid solution. When the steel is cooled thence this solution tends to break up into the condition of ferrite and cementite. If the cooling is slow this dissociation is complete, and the resultant slowly cooling steel consists of ferrite and cementite. If the cooling is rapid, then the change from solid solution into ferrite and cementite is incomplete, and the decomposition of the solid solution is more or less arrested. Different rates of suddenness of cooling give different stages of decomposition products, and to three of these stages definite names have been assigned by M. Osmond, viz., martensite, sorbite and troostite. We have the anomaly then of assigning

definite names to the decomposition products of this solid solution, and yet having no name at all for the solid solution itself. After long experience in teaching this subject to many classes of students I have become convinced that it is desirable that the solid solution itself should have a definite name to facilitate description and discussion, and for it I propose "osmondite." It is indeed wrong that the name of one whom Stead has called the greatest of metallographic micrographers, should not be represented in the nomenclature of the subject.

There is a widespread objection to the multiplication of these specific names for the different metallographic entities. This objection, however, is, I think, ill founded. It appears to me to arise from the fact that most metallographists are more familiar with chemistry than with the other branches of natural science. In chemistry specific names for chemical compounds are not necessary or desirable for the reason that the composition of the substance itself (sodium chloride, ferric oxide, etc.), is a sufficiently clear name for the substance. But in other branches of natural science in general this is not the case. In anatomy, botany, biology, petrography, and best of all, for a simile, in mineralogy it would be intolerable to try to get along without specific names. Their convenience is so great as to outweigh every possible objection.

We are in the infancy of metallography, and the number of different entities which are going to come into the discussions of metallographists, will in the future increase enormously. In many cases it will not be necessary to have specific names, either because the entity is unimportant, or because it can be sufficiently described by its chemical composition. But in the case of the solid solution of iron and carbon above the critical temperature a specific name, I am confident, is really urgently needed to facilitate teaching young metallographists and metallurgists the metallography of iron and steel. In our various institutions of learning taken together there are probably a thousand young men every year who ought to be given a general smattering of metallography of iron and steel. To adopt a specific name such as "osmondite," would greatly facilitate their studies and the labors of their teachers.

HENRY M. HOWE.

Columbia University, N. Y., January 10, 1903.



The fact that with the development of metallography the nomenclature is becoming more and more involved, induced the Council of the Iron and Steel Institute to appoint a committee to consider the matter and to ascertain whether it would be possible to take steps to make the terminology less complicated and more precise. The committee drew up a glossary, which was published in the *Journal* of the Iron and Steel Institute (1902, No. 1, pages 90-119), and it was hoped that it would tend to promote the unification of terms, the simplification of those used and the elimination of many of them. It is not, however, to be expected that the compilation of this glossary will prevent the introduction of new terms, and if the convenience of the new terms is so great as to outweigh all objections it is obvious that they deserve to be generally adopted. The well-considered proposal made in your issue of January 24 by so eminent an authority on metallography as Prof. Henry M. Howe, that the new terms "benmutic" and "osmondite" should be introduced, deserves, therefore, the most careful attention. In my opinion, however, the former of these is unsatisfactory from a philological point of view, inasmuch as the Latin prefix "bene" can only become "ben" before a vowel, and as the termination "-ic" is Greek, "benemutable" would be more correct, but undoubtedly ugly. I question whether "bene" is ever used in this sense, nor do I know of any Latin prefix so employed. "Eu" in Greek is so used again and again, and "eutectic" has a classic Greek word corresponding with it. It seems therefore that Greek cannot be avoided, and evidently "eu" is the right prefix, and "ic" which means "capable of," the right termination. What Prof. Howe should find is a suitable Greek word to represent transformation. There is, for example, the word *alloioo* ( *αλλόιοω* ) to make different, which has an adjective *alloiotikos*—changed or changeable—used by Aristotle. This would give the passable word "eualloiotic." The alternative would be to take one of the many compounds of "meta," meaning change. Aristotle uses *metablatikos*, able to produce change or subject to change. This gives "eumetabalatic." Prof. Howe's "eumetamorphic" is correct, though cumbersome. "Eumetaplastic" sounds better, but rather conveys the idea of easy to be remoulded.

To the second term "osmondite" proposed by Prof. Howe there can be no possible objection. Indeed, it would be most grati-

ifying if the name of the greatest exponent of metallography were represented in the nomenclature of the subject.

BENNETT H. BROUGH.

London, Feb. 10, 1903.

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Like Prof. Howe, I have found that the use of the term "eutectic," as applied to steel, is confusing, especially in teaching, even though it has the advantage of impressing the analogy between liquid and solid solutions. The adoption of the term "benmutic," which he suggests, is a way out of the difficulty, and the terms are sufficiently alike to suggest the similarity between substances that they represent.

I am also in favor of the term "osmondite," which Prof. Howe proposes to give to the solid solution of carbon in iron. The present descriptive nomenclature is so cumbrous that one is tempted to use the term "martensite" in default of any other, and I notice that Prof. Van't Hoff has done this in his paper "Zinn, Gips und Stahl vom physikalisch-chemischen Standpunkt." It is unnecessary to state that we are making an unwarrantable assumption in concluding that the martensite of quenched steel is identical with the solution of carbon in red hot steel before the quenching took place.

The adoption of a separate term, "osmondite," would avoid the difficulty and would be a permanent record of the debt which metallurgical science owes to M. Osmond.

ALFRED STANSFIELD.

McGill University, Montreal, Feb. 25, 1903.

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In your issue of January 24, 1903, you publish a very interesting communication from Professor H. M. Howe, entitled "Eutectic or Benmutic," in which the author suggests the use of the latter term instead of the former to designate steel made up exclusively of pearlite. This important change in our nomenclature of metallographic terms appears to me undesirable, and I shall venture to briefly state my reasons for this. The word "eutectic" was, as Professor Howe rightly says, first applied by Guthrie to that member of a series of alloys made up of various proportions of the same constituents, which had the lowest melting point. Pearlite, that is, iron containing about 0.80 per cent of carbon is not the alloy with the lowest melting point of the



iron-carbon series, and in this consideration we find at first sight, a weighty argument against the term eutectic being applied to this alloy. It should, however, be borne in mind that if pearlite is not the eutectic mixture of a molten alloy, that is, of a liquid solution, it is the true eutectic alloy of a *solid solution*. We should not lose sight of the fact that steel after it has solidified becomes a solid solution, and that during its further cooling, the various components fall out of solution in a manner singularly suggestive of the solidification of the constituents of a liquid solution, and pearlite is the true eutectic alloy of this solid solution inasmuch as it is the last one to fall out of the solution. The analogies between pearlite and eutectic alloys in general are indeed very striking. Witness the branch of curve which in the cooling curve of carburized irons, corresponds to the formation of pearlite; witness also the structure of pearlite so characteristic of all eutectic mixtures. It only differs from the eutectic alloys with which we are better acquainted in the fact that it is a constituent of last consolidation of a solid instead of a liquid solution. In view of these considerations, it appears to me preferable to retain the term eutectic for these constituents of solid solution so similar to the eutectic alloys of liquid solutions, and in order to remove the objection pointed out by Professor Howe, to slightly broaden the meaning of the word eutectic, so that it may be applied to the constituent or alloy of last *consolidation*, alike of liquid and solid solutions. By impressing students with the fact that pearlite is the eutectic alloy of a solid solution and not of a liquid solution would not the confusions to which Professor Howe alludes, be avoided?

As to Professor Howe's suggestion to give the name of "osmondite" to the solid solution of iron and carbon existing in a stable condition above the critical range of steel, all metallographists would, I believe, recognize the justice of attaching Osmond's name to a term of the nomenclature of a subject to the progress of which he has contributed more than any one else, and would welcome this addition, but has not Osmond himself proposed long ago the name of austenite for this solid solution?

ALBERT SAUVEUR.

Harvard University, Cambridge, Mass., March 2, 1903.

## METALLOGRAPHIC NOTES

**Annealing of Muntz Metal.** — Mr. E. A. Lewis published a short article on this subject, accompanied by 13 photo-micrographs. The alloy experimented upon contained 61.49 per cent of copper and 38.25 per cent of zinc. Samples of this metal were heated to different temperatures, for different lengths of time, and cooled at different speeds. Photographs of the resulting structures are shown, but no inferences are drawn. According to the author, Muntz metal consists of two constituents; "the one is a solid solution of Cu-Zn in copper ( $\text{Cu-Zn} + x\text{Cu}$ ); the other, which solidifies last, is probably a solid solution of Cu-Zn in one of the compounds of copper and zinc; it is best presented as  $\text{Cu-Zn} + x\text{Zn}$ . This latter compound is not a true eutectic alloy, but is itself made up of very small crystals; it is more easily attacked by ammonia and solvents than  $\text{Cu-Zn} + x\text{Cu}$ ." — *Journal of the Society of Chemical Industry*, January 15, 1903.

**The Lesson of the Microscope.** — Deductions from researches made in the physical laboratories, and from microscopic observations, have proved beyond question the desirability of completing hot work within certain well-defined and narrow limits of temperature, especially when steel is intended for structural uses, or for purposes where it will be shaped cold or subjected to stresses, without further hot work being put upon it.

In this respect the microscope, in the hands of those who are qualified to interpret its lessons, has confirmed the teaching of the testing room.

The great practical lesson of the microscope thus far is that steel must be given a compact and fine structure to insure safety and endurance. — *Sparks from the Anvil*, December, 1902.

**General Method for the Micrographic Analysis of Steel.** — In the proceedings of the Engineers' Society of Western Pennsylvania for December, 1902, will be found a translation of Mr. Qsmond's well-known paper entitled "General Method for the Micrographic Analysis of Steel," which was published in the *Bulletin* of the Société d'Encouragement, for May, 1895. The



students of metallography will welcome this translation, but it is to be regretted that the text translated was the original contribution of Mr. Osmond published eight years ago, instead of the extensively revised edition of this paper published by the Société in 1901 as part of a special volume entitled "Contribution to the Study of Alloys," and in which the author brings the subject to date by important revisions and additions.

**The Use of the Microscope in the Determination of the Properties of Steel.** — A stenographic report of an extemporaneous lecture, by Mr. Albert Sauveur, on the use of the microscope in the determination of the properties of steel, delivered before the Engineers' Society of Western Pennsylvania, November 22, is published in the Proceedings of that Society for December, 1902. It includes thirty-three illustrations of photomicrographs and of the necessary apparatus to conduct metallographic work. The lecturer first described the structure of various grades of steel and cast-iron as revealed by the microscope, laying special emphasis upon the close analogy which exists between the structure of steel and that of cast-iron, as had been so forcibly shown by Professor Howe. With increasing content of combined carbon, the matrix or metallic part of grey cast-iron undergoes structural changes which are identical to those observed in steel. Grey cast-iron may be regarded as composed of a steel matrix plus a certain amount of graphitic carbon, the grade of the steel composing the matrix being dependent upon the percentage of combined carbon present. Attention was then called upon the close relation which exists, on the one hand, between the structure of steel and the treatment to which it is subjected, and, on the other, between the structure and the physical properties of the metal. These considerations suggest, at least, if they do not demonstrate, the practical application of metallography. The great improvement in structure, and, therefore, in physical properties, resulting from the proper annealing of steel castings was illustrated, and the lecturer expressed his surprise that some manufacturers of steel castings could still be found who would deny the beneficial effect of this treatment, and his greater surprise that consumers of steel castings do not always specify proper annealing. The various structures imparted to the same steel by different thermal and mechanical treat-

ments were described and illustrated. It was shown that by working the steel while hot and finishing it at the proper temperature a very fine structure could be imparted to the metal; an equally fine structure could be produced by properly conducted annealing, regardless of the coarseness of the structure before annealing. It was evident, therefore, that two means could be employed for conferring a fine structure to the metal, (1) hot work suitably conducted so as to finish the implement at the proper temperature, and (2) annealing. The objection to the first method was that if the implement had a relatively large cross-section, it was not possible to finish it at a uniform temperature, the center would necessarily be hotter and would cool more slowly, and in consequence would assume a coarser structure than the parts nearer the cooling surfaces. These limitations did not exist in the case of annealing, as the piece of metal could be heated throughout evenly to the desired temperature. There was little doubt but that every finished piece of forged steel could be materially improved by annealing. The cost of the operation, however, was in many cases prohibitive. The lecturer said that many people argued that the knowledge of the structure of metals was of little importance, because if the metal was defective the testing machine would reveal it. To show how unreasonable this was, one might as well say that he did not care to know the chemical composition of the metal, since if it were defective, physical tests would detect it. The testing machine, however, merely revealed the fact that the metal was deficient in some qualities without indicating the cause, and still less, suggesting a remedy. It was just as important to know that the steel was not of too coarse a structure as it was to know that it did not contain say too much phosphorus. The lack of ductility or brittleness caused by a coarse crystallization is as objectionable and should be guarded against with as much care as the brittleness produced by too high a percentage of phosphorus. The lecturer called attention to the unreasonable attitude of some persons who with hardly any preliminary preparation took up metallography, and expected that it was going to solve at once all the troublesome problems which they had been unable to solve by other methods, and who, upon finding that their foolish expectation was not realized, blamed metallography for their failure. It reminded him of the attitude



of many steel producers toward chemistry after the chemist had succeeded in finding his way in the steel works. They had admitted him with much reluctance, but once admitted they had expected him to give an explanation of every so far unexplained phenomenon and to suggest a remedy for every evil. Upon finding him unresponsive to such demands they argued that chemistry was of little use in practical steel making, and a reaction was created which for many years hindered the advance of steel metallurgy. One could not expect that metallography would offer a solution for all the troubles of the steel maker; it was just as up-hill work in this branch of science as in any other, but if the subject were taken up in a reasonable and impartial frame of mind very few, the lecturer believed, would be disappointed.

The lecture was illustrated by numerous enlargements of photomicrographs and many apparatus were exhibited.

**Recent Publications.** — *The Manufacture and Properties of Iron and Steel*, by Harry Huse Campbell; 862 pages; illustrated. *The Engineering and Mining Journal*, New York, 1903. Price, \$5. — This is an extensively revised and enlarged edition of Mr. Campbell's well-known book on "The Manufacture and Properties of Structural Steel." The justification for the change to the present much more ambitious title might be questioned, for we find in this new edition eighty-one pages devoted to the blast furnace, eleven to wrought iron, eight to cemented and crucible steels, thirty-one to Bessemer steel, while one hundred and fifty-four pages are devoted to the manufacture of open-hearth steel. In its present form, therefore, this book is still essentially a treatise — and a most admirable one — on open-hearth steel. What the other chapters lack in quantity, however, is made up, to a great extent, by the quality, which is excellent. Mr. Campbell's book is not a text-book, for, notwithstanding an introductory chapter on "The Main Principles of Iron Metallurgy," in which the rudiments of the art are described with great lucidity, the following chapters call for such a knowledge of the metallurgy of iron as is possessed only by technically trained practical metallurgists and by advanced students. To these, however, the book should prove of very great interest and value.

The author has acquired, through an experience extending

over many years, a mastery of the practical problems with which the steel metallurgist has, every day, to deal, and to this invaluable store of knowledge he joins not only a scientifically trained mind, but one which justly appreciates the value of scientific methods, even in the domain of a purely industrial art. If we add to this a power of expressing his thoughts with much lucidity — because his thoughts are well conceived — it will be seen that the needed conditions for a valuable production were here fulfilled, and the author has given us, as he was bound to give us, a work of high merit, which will sparkle for a long time in the literature of the metallurgy of iron. It is the most valuable addition to this already rich literature, that has been made in many years.

*Hardening, Tempering, Annealing and Forging of Steel*, by Joseph V. Woodworth; 288 pages; illustrated. Published by Norman W. Henly & Co., New York. Price, \$2.50. — In this book the author describes at length the methods of hardening, tempering and annealing various implements of steel. His treatment of the subject appears to be exhaustive and is essentially practical, considerations of a scientific character being altogether absent. The purpose and scope of the book is set forth in the first paragraph of the preface as follows: "In preparing this treatise the author has had as an incitement the knowledge that there was very little information to be had on the treatment and working of steel of practical value to the general mechanic. For this reason he is convinced that a practical book on the treatment and working of the metal as modern demands necessitate, that is in regard to heating, annealing, forging, hardening and tempering processes, cannot fail to prove of interest and value to all mechanics who use tools or who are in any way engaged in the working of metals."

The book is divided into 12 chapters as follows: Chapter I. — Steel: Its selection and identification; steel for various purposes; the treatment of well known brands of steel; the effects of heat. II. — Annealing processes; the terms annealing, hardening, and tempering defined; the annealing of malleable castings. III. — The heating and cooling of steel; location of heating arrangements; the use of gas blast furnaces and heating machines; tough steel and hard steel; the difference. IV. — The hardening of steel; hardening in water, brine, oil and solutions; special processes for special steel. V. — Tempering by colors; in oil; on hot plates; by thermometer; in hot water; in the sand bath; by



special methods. VI.—Case-hardening processes; the use of machinery steel for cutting tools and the treatment of it. VII.—Hardening and tempering milling cutters and similar tools. VIII.—Hardening, tempering and straightening all kinds of small tools. IX.—The hardening and tempering of dies and all kinds of press tools for the working of sheet metal. X.—Forging and Welding. XI.—Miscellaneous kinds. XII.—Grinding.

*A Text-Book of Quantitative Chemical Analysis*, by Frank Julian; 604 pages; illustrated; published by the Ramsey Publishing Co., St. Paul, Minn. This is the work of an erudite chemist endowed with the rare gift of imparting his knowledge in a clear and effective manner. It is an admirable text-book which fills a place not occupied by any other book, not excepting Fresenius's classical work, and which should be in the library of every student of analytical chemistry. The book contains numerous original illustrations and is divided into four parts which the author describes in the following words: "In Part I., after outlining the general principles of the art, there are described the operations of solution, precipitation, etc., and the appliances commonly employed for the purposes. Following is a graded series of exercises chosen with a view to illustrate the leading principles in analysis and afford practice in the usual manipulations. They are, for the most part, simple and easy of execution, and call for only such apparatus as is commonly found in the laboratories of educational institutions. Directions are given in full detail and have been closely followed in the analysis whose results are appended.

In Part III., is considered the analytical behavior of a number of articles of commercial importance. It has been attempted to outline the most approved method for their analysis and to annotate some others that are of interest from their promise of future development or as suggesting the application of less familiar principles. Working details and criticisms have been largely omitted as they would be useless unless accompanied by particulars and precautions too voluminous for insertion here; for these there may be consulted the standard treatises on the various subjects and the references given, which are, wherever possible, to original articles or abstracts in English.

In Part IV., are presented some notes and observations relating to the principles and practice of the art in general that may be of interest to the student.

# The Metallographist

A QUARTERLY PUBLICATION DEVOTED TO THE STUDY OF METALS, WITH  
SPECIAL REFERENCE TO THEIR PHYSICS AND MICROSTRUCTURE,  
THEIR INDUSTRIAL TREATMENT AND APPLICATIONS.

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Vol. VI

JULY 1903

No. 3

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## IRON, STEEL, AND OTHER ALLOYS\*

### Introductory

I. INTRODUCTION. — Beside the general interest which we have in understanding the constitution of alloys, as giving us an intelligent view of the matter in general, we have the special reason that a knowledge of the subject promises to be of the greatest practical value in approaching the study of any given series of alloys, for instance to one seeking to learn what are the most valuable alloys of two given metals. The case reminds us of the calculus. If we have the formula of a given curve before us we can by means of the calculus discover where all the critical points of that curve will lie without going to the trouble of plotting it throughout. Somewhat so is it with the examination of the constitution of a series of alloys, say those of bismuth with tin, or antimony with copper. The constitution of such a series may be expected to vary from one end of the series to the other; but in passing thus from end to end of the series there may be important critical points, at which not only the constitution changes but the nature of that change itself changes abruptly. Such points may be called critical points for constitution. And,

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\* First chapter of Professor Henry M. Howe's new book on Alloys.



just as the calculus reveals to us the critical points of a curve of known formula, so it happens that these critical points for constitution may often be laid bare by means of a few easy experiments.

Now the importance of this lies in the fact that the critical points for constitution may be expected to be also critical points for the useful properties. If we seek ductility, we may expect to find a critical point for ductility in that part of the series where lies a critical point for constitution: there we may expect to find either a maximum or a minimum of ductility; and so with many other useful properties.

Thus it is that the methods which promise to reveal to us with relative ease the probable constitution of a series of alloys of any two metals, of learning where its critical points lie, and indeed whether it has any critical points, thereby promise to teach us where in that series we shall probably find those alloys the properties of which will differ markedly for better or for worse from those of the component metals; and indeed, whether or not we are likely to find any alloys in the series which do differ markedly in their physical properties from the component metals. The knowledge of the constitution of a series of alloys in short gives us a method of superior analysis of the problem of where to find in that series the most valuable alloys. These indications, of course, are not conclusive; indeed we have still far more to learn of their meaning than we yet know; but already they are of great value as in pointing out the part of the field most likely to be fruitful.

2. POSSIBLE COMPONENTS OF ALLOYS. — In inorganic matter we have three important classes of substances:

- (1) pure elements,
- (2) definite chemical compounds of those elements, and
- (3) solutions.

In the same way we recognize in our alloys three classes of ultimate constituents:

- (1) pure metals,
- (2) definite chemical compounds of those metals with each other, such as  $\text{AuAl}_2$ , antimonide of copper ( $\text{Cu}_3\text{Sb}_3?$ ), and antimonide of tin ( $\text{SnSb}$ ), and also to a smaller certain extent definite chemical compounds of metals with the relatively small quantities of certain metalloids, such as carbon and sulphur,

present in some alloys. In this latter class of compounds the most important is the carbide  $\text{Fe}_3\text{C}$  found in steel, and commonly called "cementite,"

(3) what are now called solid solutions of metals in each other, a term the meaning of which will be explained in § 5, p. 187. Suffice it for the present that we have in our alloys these three classes, corresponding to the three great classes into which inorganic matter in general is divisible.

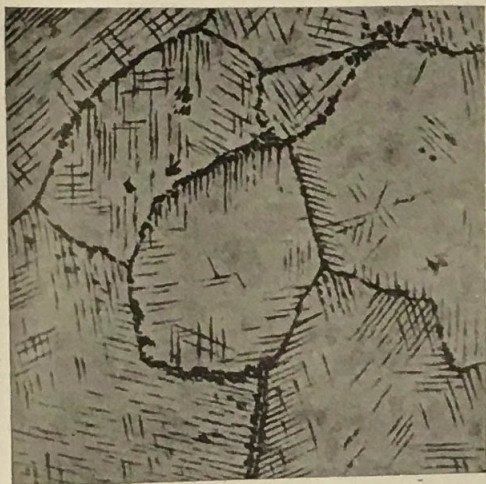


Fig. 1. Pearlite with Ferrite.

The polygons are pearlite, the network is ferrite.

(Sorby, *Journ. Iron and Steel Inst.*, 1887, I, p. 255 *et seq.*, Fig. 13.)

Any given piece of an alloy may at the same time contain substances of each of the three classes.

Here as in so many other respects the alloys remind us of the crystalline rocks, which they resemble in the general conditions of their formation. Crystalline rocks have cooled either like most of our alloys from a state of fusion, or at least from a temperature so high that the atoms present in the rock-mass have been free to arrange themselves, to combine to form definite compounds, and these compounds have been free to obey their crystalline laws.



Under the microscope we find that our rocks consist of three classes of substances:

- (1) pure metals, such as native copper, native gold, *etc.*,
- (2) definite chemical compounds, like feldspar, mica, quartz, hornblende, and
- (3) glass-like obsidians, in which the chemical elements are united, not in any definite ratio, but indeterminately.

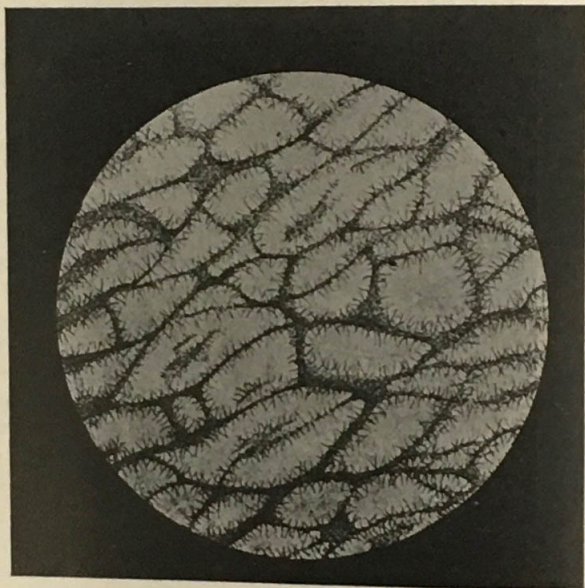


Fig. 2. Structure of Igneous Rock.

(Rhyolite from Hot Spring Hills, Pah-Ute Range)

"U. S. Geological Exploration of the Fortieth Parallel, VI,  
Microscopical Petrography," Plate VIII, Fig. 1.

Just as the particles of these different substances, the feldspar and other minerals, often exist in such minute particles that they can be detected only under the microscope, so in most cases the components of our alloys are visible only under the microscope, and often only with very great magnification, which may

sometimes have to reach a thousand diameters. Fig. 1 gives us an idea of a common type of structure among our alloys, and it will be seen that it is strikingly like that of the crystalline rocks shown in Fig. 2.

The resemblance between alloys and crystalline rocks does not stop here. Indeed we find close analogies between our metals on one hand and our crystalline rocks on the other, both of

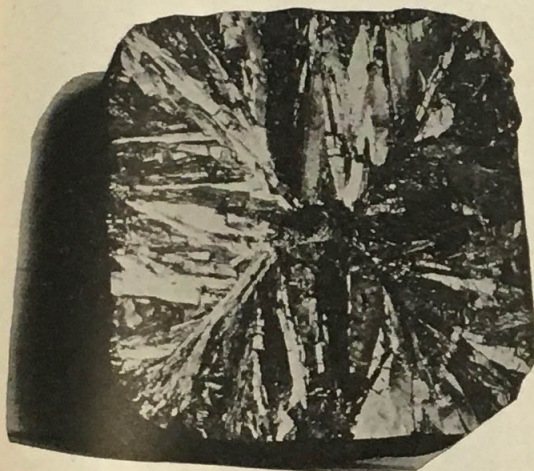


Fig. 3.

Columnar Structure in Steel Ingot,  
from the Author's Collection,  
actual size.

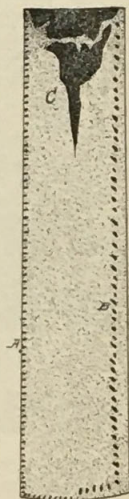


Fig. 4.

*A*, superficial  
blowholes;  
*B*, deep-seated  
blowholes;  
*C*, pipe.

which result from the gradual solidification of fused or semi-fused masses; and also between both of these classes of solids and those which, like ice, result from the solidification of aqueous solutions instead of fused masses. Let us notice some of these points of resemblance.

3. RESEMBLANCE OF METALS TO ROCK-MASSSES. — First the columnar structure familiar to us in the Palisades of the Hudson, the Giant's Causeway, and like rock-masses, forming enormous



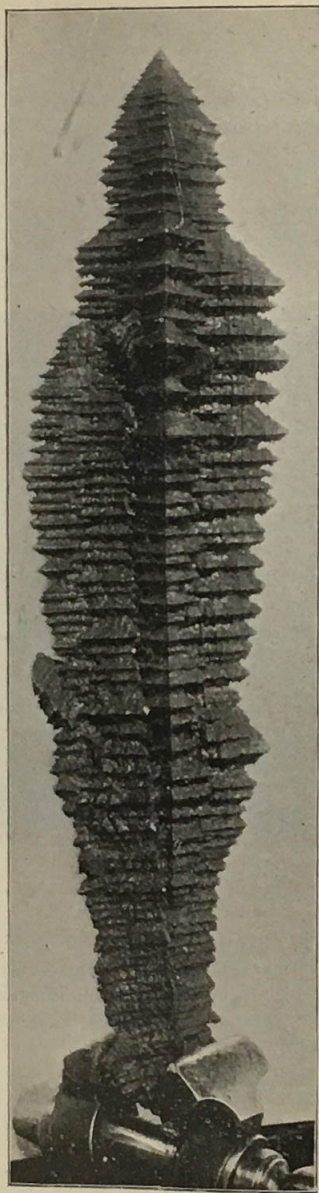
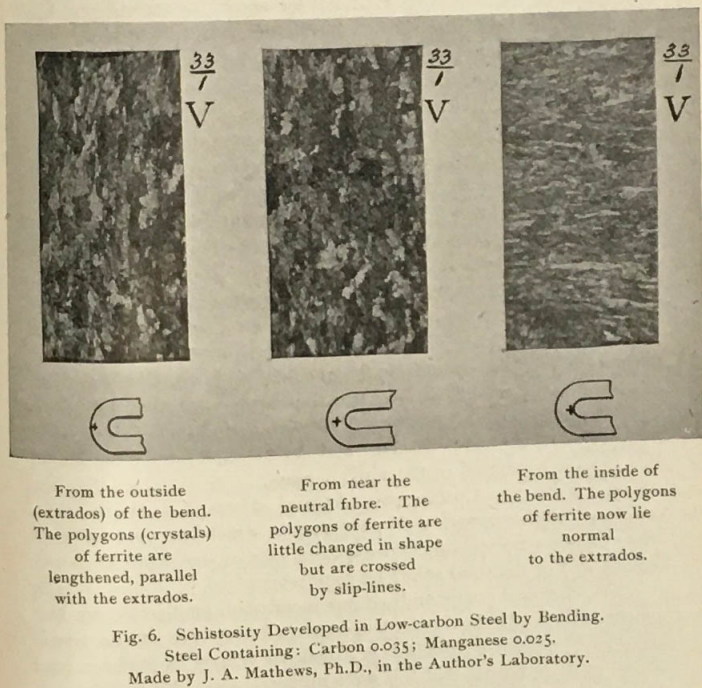


Fig. 5. Steel Crystal, about half natural size.  
From the collection of Professor Tschernoff. (*The Metallographist*, II, p. 74.)

columns, we find reproduced both in metals and in ice. The columns of the Palisades were formed during the slow cooling of the rock-mass which they form; and they stand upright, *i. e.*, with their length at right angles with their upper surface, which was the cooling surface, the surface through which the heat



escaped from them while they were cooling down and changing from a molten glass or obsidian to a solid rock. We find in slowly cooled metals this same columnar structure, with the columns standing at right angles with the cooling surface, *i. e.*, with the outer surface, as is shown in Fig. 3; and in large blocks of ice, especially in the ingots of artificial ice which we see about



the streets, we can often trace this columnar structure, with the columns at right angles with the cooling surface, *i. e.*, with the sides of the ice-ingot.

Next we find in solidified ingots of steel a contraction-cavity called a "pipe" at the upper end of the axis of the ingot (Fig. 4); and you will generally see a similar pear-shaped cavity in the upper end of the ingots of artificial ice about the streets.

Next the beautiful specimens of minerals which adorn our mineralogical cabinets generally form in the cavities or "vugs" as they are called in the rocks; beautiful crystals of iron at times occur also in the cavities in our steel ingots (Fig. 5); and in the same way we will often find most beautiful minute crystals of ice in the pear-shaped pipe of our common ingots of artificial ice.

Again, just as the crumpling of the rocks of the earth's crust produces what is known as a schistose structure, so we find such a structure (Fig. 6) in masses of iron when they have been crumpled in like manner.

Finally, gases evolved during solidification cause gas-bubbles or "blowholes" in ingots of ice and of steel, and also in glass (*A* and *B*, Fig. 4). These blowholes form at a time when the mass is still fluid enough to be pushed aside by the particles of gas evolved within it, so that these come together to form gas-bubbles; yet not fluid enough to permit these bubbles to rise by gravity to the upper surface and thus escape. So these bubbles remain entangled in the viscous mass.

4. CHEMICAL COMPOUNDS AMONG METALS. — While the condition of the particles of pure metals which we find in our alloys calls for no special comment here, that of the chemical compounds and of the solid solutions requires a word.

The chemical compounds of one metal with another do not in general follow the law of valence, so that they are of the type known as "molecular." The law of valence retains its importance, but we must recognize that it is not a universal one. We may, however, make the important generalization that obedience to the law of valence is typical of a great number of our strong chemical compounds, those in which the atoms are powerfully united, yielding a compound which differs greatly in its properties from those of its components. And it is in accordance with this general idea that even the definite chemical compounds of one metal with another, as of copper with antimony, or gold with

aluminium, or tin with antimony, as they do not follow the law of valence, are feebly combined, and that their properties differ from those of their component metals in a very much less degree than is the case with the common strong chemical compounds with which we are familiar in inorganic chemistry. The properties of water are wholly different from those of hydrogen and oxygen; the properties of common salt give no suggestion of those of either chlorine or sodium. Naturally we find no such striking difference between the properties of a definite compound of aluminium with gold on one hand, and the properties of aluminium and of gold respectively on the other.

5. **SOLID SOLUTIONS.** — What do we mean by this term? We use the word solution here to distinguish these substances from solid definite chemical compounds. We mean by solid solutions those solids which are to definite solid chemical compounds; like salt, what liquid solutions, like salt water, are to liquid definite chemical compounds, like pure water itself. We mean solids which have the essential characteristics of solutions so far as solidity itself permits.

To understand this let us ask what are the essential properties which distinguish our common liquid solutions from definite chemical compounds. In a definite chemical compound we have two essential features, (1) complete and absolute merging of the components into a new and different substance, chlorine and sodium losing their identity absolutely and forming a wholly different substance, common salt; and (2) a mathematically fixed ratio between the two components. In the case of solution, while this fixed-ratio feature is lacking, we have this same complete merging of the two substances. In a solution of water and alcohol we can neither by the microscope nor by any other means detect either the water or the alcohol; they unite to form a new substance: neither gravity nor centrifugal force separates them; the light alcohol does not rise to the surface, nor does the heavier water sink. This complete merging of their components and the absence of fixed ratio between those components, then, are the two essential characteristics of our common liquid solutions.

As the chemical forces which hold the dissolved bodies together in the new substance, the solution, are relatively feeble, naturally the properties of the solution do not differ markedly from the



mean of the properties of the two bodies dissolved in each other.

Turning to the solid state we find in our glasses a similar state of affairs, except that the glasses are solid while common solutions are liquid. The silica, lime and alkali of the glass are absolutely merged; neither the microscope nor any other means enables us to detect either silica or lime or alkali as a separate entity in the glass, so long as it remains a glass. Only when we destroy it, tearing it asunder by analysis, can we detect any of its components. We have then in the glass a chemical merging of the components; but it is in indefinite ratios. The percentage of silica or of lime can vary by infinitesimal gradations from specimen to specimen, and this variation is accompanied by corresponding progressive change in the physical properties. The change from specimen to specimen, then, both in composition and in properties, is per gradum; whereas the changes from one definite chemical compound to another, from water to hydrogen peroxide for instance, are per saltum. The glasses then have these two essentials of solutions, the substances present are (1) completely merged, but (2) in indefinite proportions.

In the same way many of our metals, as it were, dissolve in each other, and we find them in the solidified state completely merged in each other, forming alloys which differ from specimen to specimen by infinitesimal gradations, and yet the component metals cannot be distinguished in the alloy by the microscope or by any other means. The separate individual existence of each has ceased. Here then we have the essential characteristics of solutions, *viz.*, (1) complete merging of the components (2) in indeterminate proportions; and on this account we give to these substances the name "solid solutions."

And just as the properties of the liquid on, its color, density, electric conductivity, *etc.*, do not differ markedly from those of the mean of its components, so we find that the physical properties in general of those alloys which are solid solutions do not differ markedly from the mean of the properties of the metals which compose them.\*

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\* The author has found this conception of solid solutions so hard for many students to grasp that, for their sake and at the risk of seeming to most readers needlessly explicit, some further discussion, line upon line, seems desirable. When chlorine and sodium unite to form salt,

6. ISOMORPHOUS MIXTURES AND MIXED CRYSTALS. — Solid solutions are called by some "isomorphous mixtures" and by some "mixed crystals." The term "isomorphous mixtures" would naturally be suggested by the fact that the familiar cases of two definite inorganic compounds which crystallize together to form homogeneous crystals, the molecules of each substance practically entering into and completely identifying themselves with the molecules of the other, are cases in which the two substances are isomorphous, *i. e.*, when by themselves they yield crystals of the same form, *i. e.*, "isomorphous" crystals. This property then of two different substances of merging their existence completely in single crystals, is one which we associate with iso-

the two elements lose their identity completely, they are completely merged. A wholly new substance arises, salt, having little resemblance to either of its components. They have become one; they cannot be separated or distinguished from each other so long as they are united. Neither the microscope nor gravity nor centrifugal force nor any other purely mechanical force separates them or enables us to distinguish one from the other. It is this complete sinking of their individual identity, this birth of a new substance, in which neither of the component substances is distinguishable by any means whatever so long as this new substance remains undecomposed — it is this that we here call merging.

If we grind silica and magnetic iron oxide to the most impalpable powder and mix them with most extreme thoroughness, they still remain two distinct bodies, silica and iron oxide, merely intimately mixed mechanically. We can again separate them by appropriate mechanical means. Under a sufficiently powerful microscope we can still see the white particles of silica and the black ones of iron oxide. If we immerse them in a liquid heavier than the silica but lighter than the iron oxide, we separate them by gravity, the silica gradually rising to the top, while the iron oxide sinks to the bottom. Or we can separate them by means of a magnet.

But let us melt them together, and they unite and merge, they become one, and neither can now be distinguished by any means, nor separated mechanically from the other, so long as this merging remains.

Suppose that we have a room cooled to  $-40^{\circ}\text{C.}$ ; that in this we grind up ice and crystals of hydrated cupric sulphate (blue vitrol) quite as we ground up our silica and iron oxide; to simplify our ideas suppose that at this temperature the cupric sulphate and ice do not react on each other but remain separate quite as the silica and iron oxide do, and that we then mix them in a bottle with extreme thoroughness. They still constitute simply an intimate mechanical mixture; they are not merged; by sufficient magnification we can see under the microscope the transparent white particles of ice and the blue ones of cupric sulphate. By carefully tapping the bottle we can induce the heavy crystals of cupric sulphate



morphous bodies, and this association easily leads to applying the word "isomorphous" to all cases of such identification and merging. But reflection shows us that the similarity of crystalline form, even were it not only a constant concomitant but a necessary condition of this merging, is clearly not its essence. We see no conclusive reason why two metals of different crystalline forms should not completely merge. Their union could not properly be called isomorphous, but it would properly be called a solid solution.

The chief reason, however, for preferring this latter term is that it is based, not on what could at most be a concomitant or necessary condition, but on the essential properties of this class of substances, the fact that they have all the prominent essential properties of aqueous solutions save their fluidity.

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to work their way down to the bottom of the bottle, leaving the lighter particles of ice above. Now let us heat the whole over the lamp, and soon the ice melts and dissolves the cupric sulphate. Now in this solution we have no longer a mechanical mixture but absolute merging, merging as complete and as little broken up by mechanical means, as little capable of being resolved by the microscope, as the merging of our chlorine and sodium in salt or of our silica and iron oxide in our iron silicate.

Of course, we can by appropriate chemical means break this solution up; we can insert a sheet of metallic iron and precipitate the copper on it; we can break the solution up chemically; but so long as it remains a solution, the water and the cupric sulphate lack their independent existence as two separate entities mechanically mixed, and now form one entity different from either of its components. We may dispute as to the nature of this new entity, as to the nature of solutions; we may regard them as composed of ions or whatever we please; but that the solution is one whole, that in it the water and the cupric sulphate have merged their separate existence and have coalesced to form some kind of a new thing, and some one new thing, cannot be doubted. Let us leave out of sight completely our conceptions of the nature of solutions, and for the present satisfy ourselves with this idea of (1) the complete merging of the separate existence of the two components, the water and the cupric sulphate, in this new thing, the solution, (2) in indefinite ratios.

When we find that the constituents of a solid, such as the silica, lime and alkali of a glass, or the gold and silver of a silver-gold alloy, have thus completely merged and sunk their separate individual existence in this one new body, the glass or the alloy; and when we find that the composition of this solid is not in definite chemical ratio but differs by infinitesimal gradations from specimen to specimen, we say that it has the essential characteristics of a solution so far as those characteristics are compatible with solidity itself; and we call the mass a solid solution.

As to the expression "mixed crystals" it seems most unfortunate, because it is so liable to mislead, for it certainly would suggest to many a mechanical mixture. For instance, silver and gold dissolve in each other in all proportions, and the crystals of the two metals thus merged are spoken of by some as mixed crystals of silver and gold. This certainly will suggest to many that the mass is a conglomerate, a mechanical mixture of particles of pure gold and particles of pure silver, which is exactly the opposite of the truth. Indeed, whether we speak of isomorphous "mixtures" or of "mixed" crystals, the idea of mixture leads the mind rather away from than towards the conception of merging, which is the essence of the matter; while "solution" in itself suggests merging.

7. SOLID SOLUTIONS MAY BE HETEROGENEOUS. — Liquid solutions, as we know, are habitually homogeneous, because in the liquid state diffusion takes place with considerable rapidity, so that even if a solution is initially heterogeneous, diffusion tends to make it homogeneous. From the conditions under which they form, however, and from the slowness of diffusion in the solid state, we should expect solid solutions to be heterogeneous. (See § 55, p. 66.)

8. METHODS OF STUDY OF THE CONSTITUTION OF ALLOYS. — As the science of petrography was in an advanced stage when that of metallography, of the constitution and structure of metals, was in its infancy, we naturally ask why the methods so fully developed for petrography have not been applied to metallography.

We may say that the essential procedure in petrography is to recognize the minute or even microscopic crystalline grains in our rock-masses by examining them with the microscope and with the polariscope, and by finding that they have the same crystalline form, and the same effect on polarized light, as the large crystals of the same minerals with which we are already familiar in the form of our cabinet specimens. Having determined accurately the crystalline form of quartz and its effect on polarized light by the study of large-sized cabinet crystals, we are able to recognize minute or even microscopic crystals of quartz by finding that they have the same crystalline form and the same action on polarized light. Manifestly, this method is wholly inapplicable to metallography. In the first place there are very few metals and alloys of which we have cabinet spec-



imens suitable for standards with which to compare the crystalline form or other properties of the microscopic crystals which we find in our alloys.

Moreover, the metals and their alloys are opaque, so that the polariscope is wholly inapplicable. The discovery of the X-rays may have given rise to the hope that by them we could meet the difficulty of the opacity of our metals; but reflection shows that this hope was vain. For in most cases the particles of the different components of our alloys are so exceedingly minute that any individual grain of crystal would occupy but an insignificant fraction of the thickness even of the thinnest possible section, so that any given X-ray, in passing through such a section, would pass not through one individual grain or crystal of a given component, but through many different superposed crystals of all the different components which were present. In this way the shape of each one would be completely obscured and eclipsed by that of the others, and the effect of any given particle upon the X-rays would be masked by the effect of the other crystals through which that same ray would pass. There are indeed a few cases in which the individual crystals are large enough to be thus outlined by the X-rays, but even in these cases the structure can be more readily detected by microscopic study of the surface after it has been properly prepared. Thus the X-ray method is without either present or prospective value.

The chief method actually used in metallography is to correlate the results of our examination (1) of the structure as revealed by the microscope, (2) of the physical properties of individual alloys, and (3) of the physical properties of series of alloys taken as a whole. The meaning of this third method I will make clear later. We will now take up these three methods of research consecutively.

#### 9. MICROSCOPIC EXAMINATION.\*—Many of our alloys,

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\* A. Sauveur, "Microscopical Examination of Iron and Steel," *Engineering and Mining Journal*, LXIV, p. 215.

A. Sauveur, "Mechanical Uses of the Science of Metallography," *Engineering Magazine*, XVII, p. 977.

Stead, "A Practical Lesson in the Preparation of Metal Sections for Microscopic Examination," *Proceedings South Staffordshire Institute*, Session 1896-97, XII, p. 2.

Stead, "Practical Metallography," *The Metallographist*, III, p. 220.

F. Osmond, "Méthode Générale pour l'Analyse Micrographique des

as has been already pointed out, have a granitic or porphyritic structure; that is to say, like a granite they are composed of distinct grains, each grain being some one distinct entity, and of a distinct mineral species; and the different grains are of two or more different species. In the case of common granite there are grains of three different mineral species, mica, quartz and feldspar, lying side by side; and any individual grain is of some one of these species.

Now, the composition of granite taken as a whole is indeterminate, in the sense that, if we take a series of different granites and determine their ultimate composition, we find that the percentage of silica or of lime varies by irregular gradations from one specimen to the next; and the percentage of these components might vary by infinitesimal gradations from one granite to the next. But, though the composition of granite is in this sense wholly indeterminate, and though there is thus no possible composition of which we may say "This is the true composition of granite"; yet each of the several minerals which compose the granite, the mica, quartz and feldspar, is a perfectly definite chemical compound with definite physical properties.

So many of our alloys are granitic or porphyritic. They are found by the microscope to consist of grains or crystals of different definite substances. Each grain is of some one of these substances, and these different unlike grains lie side by side, like the mica, quartz and feldspar of a granite. The substances which compose these different grains in our granitic or porphyritic alloys may be (1) pure metals,\* or (2) definite chemical

Aciers au Carbone," *Bulletin Société d'Encouragement pour l'Industrie Nationale*, May, 1895. See preferably the reprint in the "Contribution à l'Étude des Alliages, Commission des Alliages," 1896-1900, p. 277.

F. Osmond, "La Métallographie Considérée comme Méthode d'Essai," *Baumaterialienkunde*, II, No. 4.

Sir Wm. Roberts-Austen, "On Photomicrography of Steel Rails," *Institution of Civil Engineers*, January 17, 1899, *Proceedings*, CXXXVI, p. 174.

Le Chatelier, "La Technique de la Métallographie Microscopique," "Contribution à l'Étude des Alliages, Commission des Alliages," 1896-1900, p. 421.

\* Pure metals are spoken of for simplicity, serving as a type; it is probable that, in most granitic alloys, the particles which may at first



compounds of two or more metals with each other in rigidly fixed atomic proportions, such as aluminide of gold ( $\text{AuAl}_2$ ), antimonide of copper ( $\text{Cu}_3\text{Sb}_3?$ ) and antimonide of tin ( $\text{SnSb}$ ), comparable with the chemical compounds with which we are familiar in common inorganic chemistry; or (3) definite chemical compounds of a metal with a metalloid, such as the iron carbide ( $\text{Fe}_3\text{C}$ ) called *cementite*, which plays a very important part in the metallography of iron and steel; *etc., etc.* One aim of microscopic examination is to ascertain whether the alloy is of this porphyritic type and consists of different distinct crystals of definite chemical composition, or whether it is of the obsidian type consisting solely of a solid solution; or whether it contains both definite minerals and also solid solutions. The further aim is to distinguish the shape, size and the properties both physical and chemical of these various components.

The first step in general is to polish the metallic mass so that a large field may be visible under high magnification. The second is to subject it to an attack, either chemical or mechanical, which will affect the different constituents in different ways, and thus enable us not only to distinguish one from another but also to determine the shape, habit and properties of each. Of these methods five deserve mention here. Of these the first three are chemical, the fourth mechanical, and the fifth partly chemical and partly mechanical. They are as follows:

(a) *Simple attack* by some solvent such as nitric acid, iodine or licorice, a method which we owe to Sorby.\* Such a method may dissolve away one component more than another, or color one component differently from another, or it may simply eat away the joints between adjacent grains of crystals, and thus disclose their shape when they are later examined under the microscope.

(b) *Weyl's Method*, which has received great development in the hands of Charpy, is to attack by means of a solvent under the influence of a very gentle electric current. Charpy uses as the two poles (1) the alloy which is under examination and (2) at times platinum and at times another alloy of a composition so

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appear to be pure metals are in fact not quite pure; but that each apparently pure metal contains, dissolved in itself, a little, and perhaps a very little, of each of the other metals present.

\* *Journal Iron and Steel Institute*, 1887, I, p. 256.

nearly similar to the first that the electro-motive force shall be very feeble, and therefore that the attack shall be extremely gentle. This gentleness is needed in all our methods. The particles which we wish to recognize and identify are so extremely minute, and often so feebly held in place, that we must use the utmost gentleness, whether in dissolving or in mechanically separating one from the other, lest we remove mechanically those components which we seek to leave untouched.

(c) *Heat-Tinting*. The different constituents may be differently colored by gently heating the polished surface, so that the more oxidizable ones become covered with oxide tints. This method has been used by Guillemin, and later by Stead,\* with most valuable results.

(d) *Relief Polishing*. Beyond these we have the mechanical methods, of which Osmond's "relief-polishing" is very important. By prolonged very gentle polishing, the softer constituents of a conglomerate alloy are worn away, leaving the harder ones standing in relief.†

(e) *Osmond's "attack-polishing"* combines the chemical and mechanical methods happily. He polishes the surface while it is exposed to a reagent which attacks or colors the different constituents differently, so that disintegration by the reagent and removal by the rubbing go hand in hand.

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## THE CASTING OF PIPELESS INGOTS BY THE SAUVEUR OVERFLOW METHOD‡

By ALBERT SAUVEUR and JASPER WHITING

**I**NTRODUCTORY. — The formation of a cavity technically called a "pipe" in the upper part of steel ingots, under the ordinary method of casting, is too well known an occurrence to

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\* *Journal Iron and Steel Inst.*, 1900, II, p. 137.

† F. Osmond, "Méthode Générale Pour l'Analyse Micrographique des Aciers au Carbone," *Bulletin de la Société d'Encouragement*, May, 1895. See preferably the reprint in the "Contribution à l'Étude des Alliages," 1896-1900, p. 277.

‡ Read at the July, 1903, Meeting of the American Society for Testing Materials, Professor Edgar Marburg, University of Pennsylvania, Secretary.



call for any description. If a "piped" ingot be cut in two longitudinally this defect will appear as shown in Fig. 1.

The great advantage which would result from being able to cast pipeless ingots is, of course, appreciated both by producers and consumers, witness the strenuous attempts which have been made to eliminate this defect. In medium-high and in high carbon steel the pipe occupies from twenty to forty per cent of the length of the ingot. In the manufacture of armor plates and of some expensive forgings as much as forty per cent of the ingot

is discarded and in this way only metal free from pipe is converted into finished product. In the manufacture of other implements, however, it is safe to affirm that a sufficient amount of metal to insure absolute freedom from pipe is very seldom rejected. Hence the danger of turning out defective material. It is our opinion that a very large proportion of the failures of steel implements of all kinds in the process of manufacture, in the testing-room, or when in use, is due to the presence within them of this defect. The production of pipeless ingots, therefore, would not only do away with the rejection of a large amount of metal, a most important item, but it would also greatly reduce the production of defective implements. The commercial value of this last consideration will, we believe, be readily granted. From the knowledge that they are not placing on the market finished implements containing this hidden flaw — a cause of much weakness and of probable eventual failure — the manufacturers should derive a feeling of

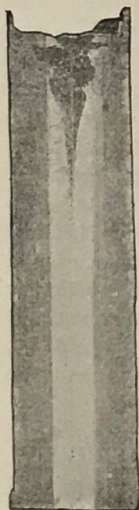


Fig. 1

security which they alone can fully appreciate.

In the production of many implements where the cost of the work required is many times greater than the cost of the metal, as for instance in the manufacture of saws and many other tools, the handling of pipeless ingots would result in a saving considerably greater than would at first appear, because the presence of a pipe is in many cases only discovered when the implement is nearly ready for the shipping-room, that is, after a large amount of expensive labor has been put upon it, and the cost of this labor is a clear loss to the manufacturer. Producers of such implements

will, we believe, readily appreciate the importance of this consideration.

**THE OVERFLOW METHOD.** — In the method now to be briefly described, it is believed that a practical means has been found for the casting of pipeless ingots. It will be sufficient for the purpose of this paper to recall in a few words the cause which induces the formation of a pipe. After molten steel has been cast into an iron mould the metal in contact with the bottom and sides begins first to solidify. After a relatively short while the top of the ingot which is exposed to the cooling action of the air also becomes solid and the ingot now consists of a rigid metallic shell holding a mass of molten steel as shown in Fig. 2. As the cooling proceeds this solid shell increases in thickness, but since steel, like most substances, undergoes a considerable contraction in passing from the liquid to the solid state, the mass of metal which when liquid was sufficient to fill the space within the solid shell, will, after it has in turn solidified, be unable to fill it and a cavity must necessarily be formed in the upper part of the ingot. It is evident, therefore, that the formation of the pipe is due to the fact that the top of the ingot solidifies while a considerable portion of the metal below it is still liquid. Once the top has become rigid the contraction of the liquid interior in passing to the solid condition must necessarily result in the formation of a cavity or pipe. By retarding the solidification of the top we should, therefore, decrease the size of the pipe and if it were possible, in a practical way, to maintain the top liquid to the very last, that is, until all metal below it has solidified, the formation of the pipe would be altogether prevented. Efforts have been made in this direction, such for instance as covering the top of the ingot immediately after casting with fuel or with molten slag, or in reheating the top of the mould. All such attempts, however, resulted only in a slight decrease in the dimension of the pipe and were accompanied by practical objections which more than offset the small gain effected.

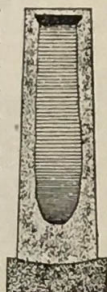


FIG. 2.

The aim of the present method is to maintain the top of each ingot liquid until all metal below has solidified and to do so without in any way interfering with the conduct of the mill operations or adding to their cost.



The method consists in so connecting a number of moulds that the molten metal can overflow from one mould into the next, while pouring is continued in the same mould until any desired number of moulds beyond have been filled.

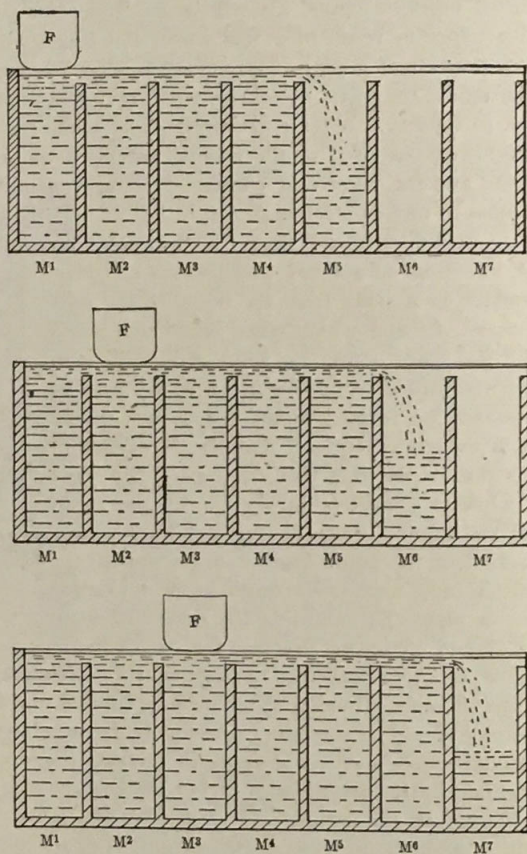


FIG. 3.

In Fig. 3 is shown a diagram representing in elevation seven moulds which we will suppose so connected that after one mould has been filled if the supply of metal be continued into it, the excess of metal will overflow into the next which will in turn be filled. To take a particular case let us suppose that we cast the

steel into mould number one and that after it has been filled we continue pouring into this same mould until we have cast into it sufficient metal to fill the next five moulds. After mould number one has been filled the metal will overflow into mould number two and when this one has been filled it will overflow into mould number three and so on until six moulds have been filled. It will be seen that by this method we maintain a flow of molten metal running over the tops of the solidifying ingots and, therefore, retard the solidification of the tops which is our desideratum. This should result in a reduction of the pipe and, if sufficient metal be passed over the tops, in the complete obliteration of this defect. The molten metal flowing over the tops of the ingots acts after the fashion of a sinking head, feeding the pipes and, therefore, preventing their formation.

It will be seen that in the example we are considering ingot No. 1 will have enough metal cast upon it to fill five moulds, ingot No. 2 enough to fill four moulds, ingot No. 3 enough to fill three moulds and so on to the last ingot which had no extra metal passed over its top. The amount of molten steel flowing over the tops of these ingots decreases, therefore, as we pass from the first to the last ingot, and it should be expected that the effectiveness of the method in eliminating the pipe should be greatest in the case of ingot No. 1 and should then gradually diminish.

SEGREGATION. — It is well known that the segregation of impure metal in the upper part of steel ingots, that is in the piped portion, is due to the fact that the impurities present in the metal, especially the phosphides, sulphides and carbides, are more fusible than the metal itself, and have therefore a tendency to collect in the portion of the ingot remaining molten longest. In the overflow method these impurities should naturally rise to the very top of the ingot and should then be carried away by the flow of molten metal to be discharged into the next empty mould. Here however they should be diluted so largely by the metal from the crucible or ladle as to have but a very slight effect upon the average composition of the ingot. We have not as yet had the opportunity of testing the effect of the method upon the segregation of impurities, but from these theoretical considerations it should naturally be anticipated that if it does not prevent it altogether it should greatly diminish it.



THE OVERFLOW METHOD AND THE CASTING OF CRUCIBLE STEEL INGOTS. — Numerous experiments were conducted with crucible steel ingots in order to ascertain the practical value of the overflow method. The results obtained in casting six ingots, after the manner just described, are shown in Fig. 4, which is the reproduction of a photograph of the broken tops of these ingots. These ingots measured  $3\frac{1}{2}'' \times 5\frac{1}{2}'' \times 22\frac{1}{2}''$  and weighed one hundred pounds. The steel cast contained from 0.9 to 1 per cent carbon and was produced in a regenerative crucible furnace.

Ingots cast in the usual manner had a pipe extending about eight inches downward, and this was found to be the average length of the pipe for the grade of the steel and the size of the ingots experimented upon.

Turning our attention to the ingots cast by the overflow method, it is seen that No. 1 and No. 2 ingots are absolutely free

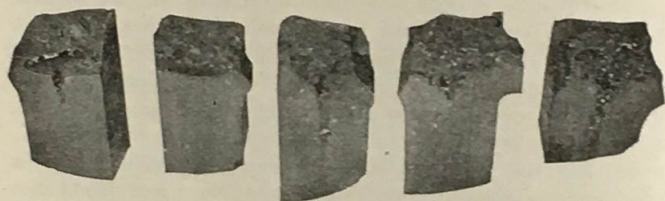


Fig. 4.

from pipe. They are solid masses of metal to the very top. The metal which was cast upon these ingots had caused their tops to remain liquid until the metal below it had become solidified, preventing thereby the formation of a pipe. No. 3 ingot shows a small cavity measuring about one inch, while No. 4 has a two-inch pipe, and No. 5 a four-inch pipe. No. 6, which is not shown here, was not of course materially improved. These experiments were repeated a number of times and the results obtained were in every case very similar.

The results which had been anticipated from purely theoretical considerations are, therefore, fully confirmed. From the appearance of No. 5 ingot we infer that the passing over the top of an ingot of sufficient metal to fill one more mould by causing it to overflow into it will reduce the length of the pipe some fifty per cent, while the passage over the molten top of an ingot of

enough metal to fill four or at the most five more moulds will result in the complete elimination of that cavity.

In Fig. 5 are shown a number of pipeless or nearly pipeless ingots obtained by the overflow method.

In casting six ingots then in the manner just described, while the two first ingots will be pipeless, the remaining ingots will have small pipes of increasing length. It may be readily conceived, however, that the overflow method may be conducted in a more continuous manner, so that enough metal will flow over the tops of all the ingots to make them pipeless, namely, in the present case, enough metal to fill four or five additional moulds. If for instance, returning to Fig. 3, after having filled six moulds by pouring in No. 1, the pouring be shifted to No. 2, and enough metal be cast to fill mould No. 7, and then the pouring be shifted

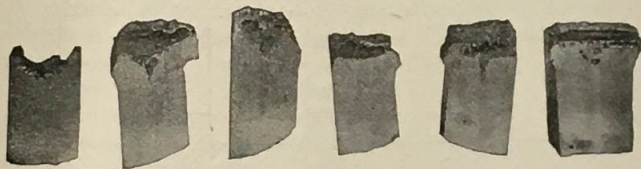


Fig. 5.

to No. 3 and enough metal be cast to fill No. 8 and so on, it is evident that the necessary amount of metal will flow over the top of each ingot to prevent the formation of a pipe.

Instead of shifting the pouring ladle or crucible from mould to mould it is evident that this receptacle may remain stationary and that the moulds themselves may be brought in turn under the ladle, by being mounted on cars or by any other suitable means, and this manner of working the continuous method is not unlikely to prove the most attractive of the two. The metal from the various converters of a Bessemer plant might be poured into a stationary mixer and from it cast into the moulds mounted on cars and in fast running American mills a continuous flow of mixer metal might be maintained, making the casting process truly continuous. Among the advantages resulting from such method might be mentioned the elimination of the "butts" resulting from the frequent partial filling of the last mould inevitable



in intermittent casting methods and which is a source of considerable loss to some steel makers, and notably to rail makers.

It is quite obvious that the amount of metal which must flow over the top of an ingot in order to make it pipeless will vary with the size of the ingot, the temperature of the metal, the rate of cooling, the composition of the steel, etc. In each mill the most desirable manner of conducting the overflow method will have to be ascertained by a few preliminary experiments.

CONNECTION BETWEEN MOULDS. — A suitable connection between moulds had to be devised and this important point was finally solved with complete success by using the moulds shown in Fig. 6. They will be seen to be three-sided moulds, each mould moreover being in two sections for convenience in stripping.

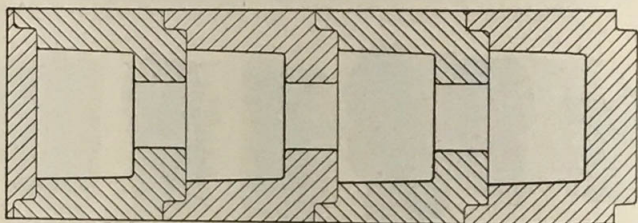


FIG. 6.

When these moulds are properly assembled and fastened together, each two adjacent moulds have a common wall between them at the upper part of which a groove is provided for the overflowing of the metal from one mould into the next. These moulds have given very good satisfaction. They are, if anything, less expensive than the ordinary style of moulds used in crucible steel manufacture.

TEEMING. — It was found undesirable to teem directly into the mould as in doing so the metal is agitated to too great a depth during the teeming to allow of the quiet, undisturbed cooling desired. To avoid this disturbance caused by the fall of the metal into the mould, a receptacle was used, as shown in Fig. 7, and which consists of a crucible-like appliance provided with a false bottom. Each bottom is provided with one hole of suitable size. This crucible rests upon the mould into which it is desired to teem and the steel is poured into it in the usual manner. The

metal flows into the lower chamber and from it into the mould. The bottom hole being larger, the lower chamber is constantly being drained during the casting. In this way the fall of the metal is broken and the steel in falling into the mould produces very little agitation of the molten top.

The overflow method is now being installed\* at the steel works of the Simonds Manufacturing Company of Chicago where the experiments were conducted.

THE OVERFLOW METHOD AND THE CASTING OF BESSEMER AND OPEN-HEARTH STEEL INGOTS. — It was quite natural to first apply the method to the casting of crucible steel ingots, but experiments are now under way to ascertain its value in casting Bessemer and open-hearth ingots.

We desire here to express our warmest appreciation to the management of the Simonds Manufacturing Company, which not only allowed us to make the tests in their mill, but rendered us invaluable services through their intelligent coöperation and good will. We are especially indebted to Mr. Daniel Simonds, president of the Simonds Manufacturing Company, to Mr. L. E. Howard, Superintendent, to Mr. W. C. Bird, Chemist, and to Mr. W. G. Merriman, Superintendent of the steel works.

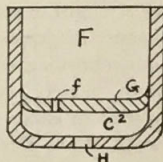


FIG. 7.

## ON THE CONSTITUTION OF CAST IRON\*

By HENRY M. HOWE

OF the constitution of steel, and of the relation between its composition on one hand and its microscopic constitution and its physical properties on the other, we have already accumulated much knowledge. It appears to the writer that this knowledge offers a point of vantage from which to begin the study of the like relation between the composition and properties of cast iron. It is true that an important class of writers virtually insists that this knowledge must not thus be used, apparently holding that the

\* Read at the June, 1902, Meeting of the American Society for Testing Materials.



very act of conferring a distinct name, cast iron, upon the iron-carbon compounds rich in carbon, has in some way operated to debar investigation along this line. But as these writers have offered no reason in support of their injunction, save their own undoubted authority, and as the writer is no respecter of authorities, he proposes in the present paper to resume this banned mode of study.

Regarding cast iron as composed of two distinct parts, (1) its graphite, and (2) the remainder of the mass, which is metallic and may for brevity be called the matrix to distinguish it from the graphite, I advanced, in an earlier paper,\* the principle that the strength of the whole mass ought to be (1) inversely as the quantity of graphite, which I regarded as necessarily a weakening and embrittling substance, and (2) dependent on the composition of the matrix, increasing or decreasing as this approached or receded in either direction from the composition of the strongest steel. While this composition, which should give the strongest matrix, should depend greatly on the quantity of the different elements such as silicon, phosphorus, manganese and sulphur, yet, were these constant, then the strongest matrix should be that with 1 or 1.20 per cent of carbon, because that is the strongest steel, the strongest iron-carbon compound, as appears from many investigations.

In the present paper I propose to develop these ideas somewhat, and in particular to test them by presenting evidence as to the truth of two corollaries from them.

Graphiteless steel and graphiteless white cast iron seem to form a single continuous series, which we may call the pearlite series. That is, they seem to consist either (1) of pearlite alone, or (2) of pearlite plus ferrite, or (3) of pearlite plus cementite, according to whether they contain (1) 0.80 per cent of carbon, or (2) less or (3) more than this quantity.

This has been shown to be true for our common carbon steels; and the present work assumes, for simplicity, that it is true for the whole range of graphiteless steels and graphiteless white cast iron. Naturally, this hypothesis should in time be tested.

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\* "The Constitution of Cast Iron, with Remarks on Current Opinions Concerning It." *Trans. American Inst. Mining Engineers*, XXXI, p. 318, Feb., 1901. — *The Metallographist*, Vol. IV, p. 177.

Table I gives the microscopic constitution of this pearlite series on this hypothesis.

TABLE I. — *Theoretical Constitution or Microstructural Composition of the Pearlite Series, Slowly Cooled Steel and White Cast Iron.*

Name		Carbon Per Cent	PROXIMATE COMPOSITION Per Cent				
			Pearlite	Excess* Ferrite	Excess* Cementite	Total Ferrite	Total Cementite
STEEL	Low Carbon	0	0	100	0	100	0
		0.10	12	88	0	98.5	1.5
		0.20	25	75	0	97.0	3.0
	Medium Carbon	0.30	37	63	0	95.5	4.5
		0.40	50	50	0	94.0	6.0
		0.50	62	38	0	92.5	7.5
		0.60	75	25	0	91.0	9.0
		0.70	87	13	0	89.5	10.5
	High Carbon	0.80	100	0	0	88.0	12.0
		0.90	98	0	2	86.5	13.5
		1.00	97	0	3	85.0	15.0
		1.10	95	0	5	83.5	16.5
		1.20	93	0	7	82.0	18.0
		1.30	91	0	9	80.5	19.5
		1.40	90	0	10	79.0	21.0
		1.50	88	0	12	77.5	22.5
		1.60	86	0	14	76.0	24.0
		1.70	85	0	15	74.5	25.5
		1.80	83	0	17	73.0	27.0
		1.90	81	0	19	71.5	28.5
WHITE CAST IRON		2.00	80	0	20	70.0	30.0
		2.10	78	0	22	68.5	31.5
		2.20	76	0	24	67.0	33.0
		2.30	74	0	26	65.5	34.5
		2.40	73	0	27	64.0	36.0
		2.50	71	0	29	62.5	37.5
		2.75	66.75	0	33.25	58.75	41.25
		3.00	62.5	0	37.5	55.0	45.0
		3.25	58.25	0	41.75	51.25	48.75
		3.50	54.0	0	46.0	47.5	52.5
		3.75	49.75	0	50.25	43.75	56.25
		4.00	45.5	0	54.5	36.25	63.75
		4.50	37.0	0	63.0	32.5	67.5

\* "Excess Ferrite" means the ferrite in excess over the pearlite ratio, or in other words the free or structurally free ferrite which does not form part of the pearlite. "Total Ferrite" means the sum of this excess ferrite plus that contained in the pearlite. So, *mutatis mutandis*, with "Excess Cementite" and "Total Cementite."



Fig. 1 sketches the physical properties of this series in a general way. The lines here given must be taken simply as indicating roughly the normal properties of the materials. It should of course be applied with great caution. For instance, we know that the strength of steel can be more than doubled by varying the rate at which it is cooled. Other causes also change its strength greatly.

Gray cast iron, together with those high-carbon steels which contain graphite (though this is usually in very small quantity), form a second series, which we may call the graphito-pearlite series. That is to say, they consist essentially of (1) their matrix, which is some one member of the pearlite series, plus (2) their graphite.

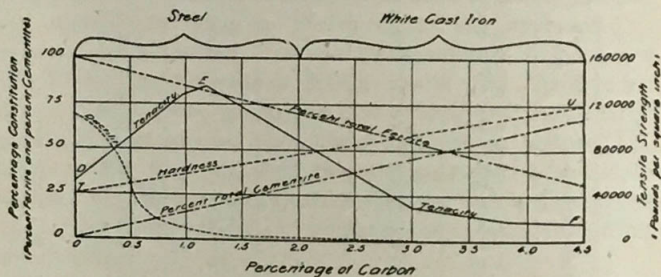


FIG. 1. Physical Properties and Assumed Microscopic Constitution of the Pearlite Series. Graphiteless Steel Slowly Cooled, and White Cast Iron

NOTE. By "Total Ferrite" is meant both that which forms part of the pearlite and that which is in excess of the pearlite, taken jointly. So with the "Total Cementite."

By their "matrix" I mean the whole of their ferrite and cementite, i.e., their metallic part. This forms a sort of metallic matrix, in which is imbedded the wholly non-metallic graphite practically as a foreign body. This matrix, in that it consists, according to our hypothesis, of ferrite and cementite, is identical in composition and constitution with some one member of the pearlite series, because every member of that series is thus composed of ferrite and cementite. Thus these graphitiferous steels and cast irons, as has just been said, consist, according to our hypothesis of (1) a metallic matrix of ferrite and cementite, identical in each case with some one member of the pearlite series, and (2) their graphite

*Constitution and Properties of Mottled and Gray Cast Iron, the Graphito-Pearlite Series.* — In the paper already referred to I made a first attempt to reconcile the constitution and properties of the pearlite series. That task was a relatively simple one; the parallel task in our present case of the graphito-pearlite series is more difficult, perhaps not so much because we as yet only conjecture the constitution of this series and because our knowledge of its properties is extremely fragmentary, as because of the greater complexity of its constitution. Table I shows that, as regards the relation between composition and constitution, we may for our present purpose assume that the pearlite series has but a single variable, the carbon-content; it is, as it were, a single line, each percentage of carbon corresponding to a fixed and definite percentage of ferrite and of cementite respectively. The graphito-pearlite series on the other hand, has two variables, the carbon-content, and the distribution of that carbon between the conditions of free graphite and of combination as cementite.\* The series thus is not a single line but a field, since for each percentage of total carbon the distribution between the graphitic and cementite states varies widely; and with each of these variables, total carbon-content and carbon-distribution, the constitution varies.

Let a single instance of each of these variations serve as an example, adopting our present hypothesis:

(1) *Total Carbon Constant, Graphite Varies.* — A cast iron with 4 per cent of carbon may have all, or none, or any intermediate part of that carbon in the form of graphite. If the whole 4 per cent is graphite, then the cast iron as a whole is a conglomerate of (1) that 4 per cent of graphite plus (2) a metallic matrix forming 96 per cent of the whole mass, and itself consisting of pure ferrite. If, on the other hand, only 1 per cent of that carbon is graphite and the remaining 3.00 per cent is combined as cementite, then the cast iron as a whole is a conglomerate of (1) 1 per

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\* In the present paper I discuss the constitution and properties of only slowly cooled material supposed to consist of pearlite and ferrite or cementite, together with graphite. The carbon present, then, will be in every case either in the condition of graphite or of combined, i.e., cementite carbon. "Combined carbon," then, in this article refers to cementite carbon, which for uniformity I have spoken of throughout as combined carbon.



cent of graphite, and (2) a metallic matrix forming 99 per cent of the whole mass, and itself containing  $(4 - 1) \times 100 \div 99 = 3.03$  per cent of carbon, and therefore by Table I consisting of 54.55 per cent of ferrite and 45.45 per cent of cementite, or say 55 and 45 per cent. To recapitulate this:

TABLE 2

Name of Cast Iron	Per Cent of Carbon	CONSTITUTION			
		Graphite Per Cent	Metallic Matrix Per Cent	Constitution of Matrix	
				Ferrite Per Cent	Cementite Per Cent
Very open gray . . . . .	4	4	96	100	0
Mottled . . . . .	4	1	99	55	45

(2) *Graphite Constant, Total Carbon Varies.* — Let us next assume that there is 1 per cent of graphite, and that the total carbon varies from 3 to 4 per cent. With 3 per cent of carbon, the constitution is (1) free graphite 1 per cent, (2) metallic matrix 99 per cent, itself containing  $(3 - 1) \times 100 \div 99 = 2.02$  per cent of carbon, and, therefore, by Table I consisting of ferrite 69.7 per cent, cementite 30.3 per cent, or say 70 and 30 per cent.

With 4 per cent of carbon, on the other hand, the constitution is (1) as before 1 per cent of free graphite, and (2) as before 99 per cent of metallic matrix, consisting of 55 per cent of ferrite and 45 per cent of cementite. To recapitulate this:

TABLE 3

Name of Cast Iron	Per Cent of Carbon	CONSTITUTION			
		Graphite Per Cent	Metallic Matrix Per Cent	Constitution of Matrix	
				Ferrite Per Cent	Cementite Per Cent
Mottled . . . . .	3	1	99	70	30
Mottled . . . . .	4	1	99	55	45

*Correspondence Between the Constitution and Properties of the Series.* — Rather than seek to cover this whole area of the graphito-pearlite series, let us consider a single line in that area; for the principles which apply to that line should apply to the area as a whole. Let us select for our line that of total carbon 4 per cent, and graphite varying from 0 to 4 per cent. This is perhaps the most useful line for our study, because it passes about through the average of our commercial pig irons, a large proportion of which contain not far from 4 per cent of carbon, i.e., are approximately saturated with carbon in the blast-furnace. These irons, or the most important of them, then, differ relatively little in their total carbon-content, but they differ very greatly in the distribution of that carbon between the conditions of graphite and of combined carbon.

Let us see how far the properties of such a series of cast irons, so far as we know them, agree with our hypothesis, that each member of this series is essentially a conglomerate of (1) the corresponding member of the pearlite series as a metallic matrix, and (2) graphite.

To this end let us try to forecast the properties of this series of cast irons all containing 4 per cent of total carbon, but with the distribution of that carbon between the state of graphite and that of cementite varying. All the carbon that is not in the state of graphite is assumed to be in the state of combined carbon in the cementite of the matrix; which assumption fixes the constitution and composition of the matrix for every assumed percentage of graphite.

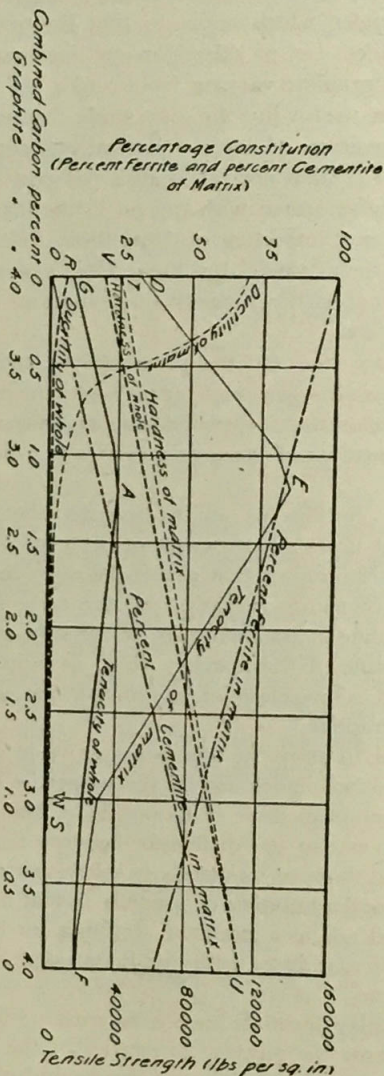
In order to arrive at this forecast, we in effect ask, in what direction and how much should the properties of the cast iron as a whole differ from those of its matrix? And we answer this on the assumption that the difference between the properties of the cast iron and those of its matrix is solely that due to the presence of a mechanical admixture of graphite in that matrix.

We next ask, as a means of verifying our hypothesis, how far does this forecast agree with our knowledge of the properties of such cast irons?

Fig. 2 may facilitate such a forecast. First the lines which in Fig. 1 show the physical properties of the pearlite series and those showing the proportion of ferrite and of cementite in that series are here reproduced, with the same lettering as in Fig. 1.



Name of matrix	Low carbon steel	Medium carbon steel	High carbon steel	White cast iron
Name of the cast iron f.c., of the whole	Very open gray or very graphitic cast iron		Close gray cast iron	Mottled cast iron
				White cast iron



Strength	Weak	Strongest	Weak
Ductility	Least brittle		Most brittle
Hardness	Softest	Soft	Hardest
strength and ductility	shock	moderate shock the iron is suited to most engineering purposes	no shock unless metal is strongly supported
Be-cause of its hardness	the iron is suited to uses in which it will have to undergo or resist	much machining in preparation, but little abrasion in use	moderate machining in preparation, not excessive abrasion in use

FIG. 2. Physical Properties and Assumed Microscopic Constitution of Cast Iron containing 4 per cent of Carbon, as Affected by the Distribution of that Carbon between the Combined and Graphitic States.

These lines are supposed to show the constitution and properties of the matrix, for varying composition, i.e., for varying carbon-content, of that matrix on the assumption that the properties of the matrix itself, its tensile strength, ductility and hardness, are the same as those of a graphiteless steel or a graphiteless white cast iron which has the same carbon-content and hence the same microscopic constitution as the matrix. For instance, the mottled cast iron of the second line of Table 3 is represented by ordinate  $W$  of Fig. 2. Table 3 shows us that the constitution of its matrix is ferrite 55 per cent, cementite 45 per cent. Turning now to Table 1 we find that this is the constitution of white cast iron containing 3 per cent of total carbon, all of which is combined as cementite. Accordingly, the lines in Fig. 2 which give the constitution of this matrix ("Per cent Ferrite in matrix" and "Per cent Cementite in matrix") cut this ordinate  $W$  at 55 per cent and 45 per cent respectively. Next, Fig. 2 assumes that the properties of this matrix itself, as distinguished from the cast iron which contains it are the same as the properties of such a white cast iron with 55 per cent ferrite and 45 per cent cementite; hence the line "Tenacity of matrix" cuts ordinate  $W$  at the same height as that at which it cuts the 3 per cent carbon ordinate in Fig. 1; and so with the lines "Hardness of matrix" and "Ductility of matrix."

These three lines, then, give us the tenacity, ductility and hardness on the present hypothesis, of the matrix of each cast iron of this series shown in Fig. 2, as a basis for forecasting the properties of the cast irons themselves regarded as a conglomerate of (1) their matrix and (2) graphite; the properties of this conglomerate, i.e., of the several cast irons each taken as a single conglomerate whole, are represented by the lines "Tenacity of whole," "Ductility of whole" and "Hardness of whole." The forecast which will here be made will be a first attempt to determine the position of these three lines, as inferred from our hypothesis.

Above the diagram is a line showing the trade name or grade of the cast iron as a whole, and above it a second line showing the nature of the matrix by what we may call its trade name. In other words, this line gives the trade names of the different classes of steel or cast iron to which the matrix corresponds in constitution, in different regions of this series. For instance, the words



"Medium carbon steel" in this line mean that the matrix of the cast irons beneath these words has the same ultimate chemical composition and therefore, on our hypothesis, the same microscopic constitution, i.e., the same percentage of ferrite and cementite, and also the same physical properties, which medium carbon steel has.

Below the diagram are given the observed properties of the different kinds of cast iron here represented, and the kinds of uses to which they are fitted.

#### ATTEMPT AT A FORECAST.

Let us first ask, "How should the interspersing of flakes of graphite affect the properties of the several members of the pearlite series?" Or, in brief, "Should the tenacity, hardness and ductility lines of the graphito-pearlite series lie above or below those of the pearlite series, and how far above and below?"

At the right end of the diagram, where graphite is zero, each line for the properties of the cast iron as a whole should touch the corresponding line of the matrix; i.e., the properties of the matrix and of the whole are identical because the matrix is the whole. This fixes the right-hand end of each line.

Let us consider the position of the remainder of the line for tensile strength, and then for ductility and hardness.

*Tensile Strength.*—As graphite is itself a very weak substance, interspersing flakes of it through our metallic matrix should be qualitatively like setting weak links in a strong chain; or, since the flakes of graphite are not continuous, like here and there notching or sawing part way through certain links of a chain. It needs no persuasion to show that these continuity-destroying flakes should weaken the mass, and that the weakening effect should increase with their quantity. We admit then that the tenacity line for the cast iron must throughout lie beneath that of the matrix.

How far below  $D$  will its left-hand end lie? From our general knowledge of the facts we may put the strength of this cast iron, i.e., the position of  $G$ , at 12,000 pounds per square inch, as the tensile strength of a very weak cast iron. At first we should suppose that as, on passing to the right, the proportion of graphite decreases continuously and regularly, so should its

influence, and so in fine should the gap between the tenacity line for the cast iron and that for the matrix decrease regularly; that is to say, the distance between the two lines should be a regularly decreasing percentage of the ordinate of line *DEF*: in short, we should at first expect a *regularly decreasing-percentage gap*. But this assumption would be too far wrong, as appears clearly even on the superficial examination here possible. Let us look a little into this question.

The weakening effect of graphite is due primarily to its breaking up the continuity of the metallic matrix. Assuming first, for the moment, that the influence of the graphite is proportional to the area of the flakes of graphite, and that the area is proportional to the percentage of graphite, and assuming as we have, that the left-hand cast iron of our series with 4 per cent of graphite and no combined carbon, has a tensile strength of 12,000 pounds per square inch against 50,000 pounds in case of pure ferrite, then in this case the weakening effect of our 4 per cent of graphite is  $50,000 - 12,000 = 38,000$  pounds per square inch or 76 per cent. Supposing the influence of graphite to remain proportional to this, what should the strength of the cast iron with matrix of composition *E* be? The percentage of graphite in this iron is 2.80. The weakening effect of this quantity of graphite should be  $76 \times 2.80 \div 4.00 = 53$  per cent. The tensile strength of the matrix itself should, from Fig. 2, be 135,000 pounds per square inch. As the tensile strength of the cast iron as a whole is to be 53 per cent below that of the matrix, it should be  $135,000 (100 - 53) \div 100 = 63,500$ .

This, then, is the first result of our attempt to test our hypothesis. Is this result sufficiently near the truth to support the hypothesis? Manifestly not. From all our knowledge of the subject, it is highly improbable that cast iron of this composition, combined carbon 1.20 per cent, graphite 2.80 per cent, has so great tensile strength.

In view of this, we next consider whether in making our calculation, we have not omitted some important consideration, which should affect our result, and bring it closer to the truth. Many such considerations may exist; three certainly suggest themselves. Of these the first two have to do with the rate at which the influence of the graphite increases, while the third has to do with the variations in the effect of the graphite due to its



changing composition and properties of the matrix itself. We will now take these up consecutively.

*The Influence of the Graphite Increases Less Than Proportionally to its Percentage.* — For simplicity I have thus far assumed that the influence of the graphite on tensile strength ought to be proportional to the percentage of that substance. This might be true in case the dimensions of the individual flakes of graphite remained constant, so that a cast iron with 4 per cent of graphite differed from one containing 2 per cent simply in having twice as many flakes, but of the same area and thickness. If, however, the increase in the quantity of graphite was accompanied by a change in the size or shape of the individual flakes, then, while the influence of the graphite should still increase as the quantity of graphite increased, so that the influence of the graphite should be as some power of its percentage, yet this power should not be unity.

Two considerations go to show that this power should be less than unity, or in other words, that the first small quantity of graphite ought to have a more powerful effect, per cent for per cent, than later additions. These considerations are as follows:

If we were to assume that, as the percentage of graphite increased the number of flakes of graphite remained constant, and that each flake or sheet grew simultaneously both in thickness and in width, so as to preserve the initial relation between its thickness and its area; then the area of the flakes should increase as the two-thirds power of the percentage of graphite. Now it might be reasonably held that the weakening effect of these flakes of graphite should be more nearly proportional to the area of the several flakes than to their volumes; and hence that, on this assumption, the influence of the graphite should increase more nearly as the two-thirds power of the percentage of graphite than directly as that percentage.

As it is probable that, as the graphite increases, there is an increase both in the number of flakes and in the area of certain of them, this consideration points to a power between two-thirds and unity.

The second of our three considerations is that, once our matrix has been weakened by the notching effect of the presence of a certain given quantity of graphite, a further increase in

this notching through an increase in the percentage of graphite should not have a proportionally great effect. This may be made clearer by considering the effect of a like increase of notching on the strength of a strip of cloth under tensile stress.

Thus, if we were to cut above the notch *A* in Fig. 3 a second notch just like it, this second notch should not materially add to the weakening effect caused by the first. Nor, if a second notch should be cut at the opposite edge of the cloth, on the same horizontal line as *A*, is it clear that this second notch would add greatly to the effect of the first. It would be like

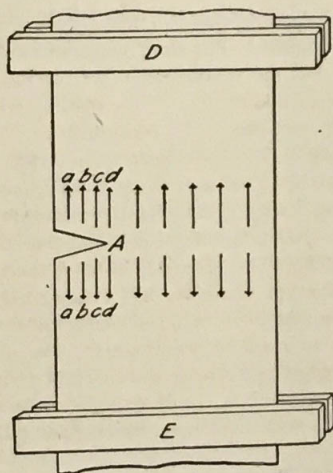


FIG. 3. Effect of Notching on Tensile Strength

filing on one side of a given link in a chain a notch exactly like that one already filed on the other side of that link. The second notch would certainly weaken the chain somewhat, but it would not double the effect of the first notch. Here, then, is a second reason for holding that the weakening influence of the notching caused by the presence of flakes of graphite should increase according to some power less than unity, of the percentage of graphite itself.

As the immediately preceding consideration pointed to a power between two-thirds and unity, and as this last considera-



tion makes for a further lowering of the power, let us adopt temporarily two-thirds as this power, and again calculate on this new assumption the tensile strength of cast iron with matrix *E*. Doing this we find that the presence of 2.80 per cent graphite lowers the tensile strength by 60 per cent, which gives a calculated tensile strength of 54,000 pounds per square inch.\*

But this is still too high; it is not probable that cast iron of this composition, combined carbon 1.20 per cent, graphite 2.80 per cent, has such great strength. So we turn to our next consideration, based on the changing constitution and properties of the matrix.

*Influence of the Properties of the Matrix on the Weakening Effect of the Graphite.*—The third consideration to which attention is asked is that the weakening effect of the graphite should increase with the carbon-content of the matrix, which the graphite weakens through breaking up its continuity.

This breaking up of continuity is in effect like nicking or notching our matrix. But all our experience shows us that the effect of notching varies greatly with the nature of the material notched. In two like rods, one of oak and the other of glass, file a like sharp notch. The strength of the oaken rod is hardly affected, while that of the glass rod is lessened out of all proportion to the quantity of material removed by filing, so that the rod is easily snapped by the fingers. The effect is like that of notching one edge of a strong roll of cloth (Fig. 3). Suppose that such a piece of cloth is firmly gripped at its two ends by the frames *D* and *E*, and that these frames are pulled away from

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\* The details of the calculation are as follows: We have already calculated that the weakening effect of 4 per cent of graphite was 76 per cent, i.e., that it brought the tensile strength of the cast iron as a whole 76 per cent below that of its matrix. Following the assumption that the weakening effect of the graphite is as the two-thirds power of the percentage of graphite, the weakening effect of the 2.80 per cent of graphite corresponding to point *E* should be found by the proportion:

$$4^{\frac{2}{3}} : 2.80^{\frac{2}{3}} = 76 : x = 60 \text{ per cent.}$$

The tensile strength of this cast iron with 2.80 per cent of graphite and 1.20 per cent of combined carbon should be 60 per cent less than that of its matrix; this we have already seen has a tensile strength of 135,000 pounds per square inch; so that the tensile strength of this cast iron should be  $135,000 \times 40 \div 100 = 54,000$  pounds per square inch.

each other; the cloth comes under tensile stress, which we may assume is evenly distributed, and represented by the arrows. Because of the notching, the material at *A*, which, but for the notching, would have to resist only stress *dd*, now has in addition to resist a large part of stresses *aa*, *bb* and *cc*. It yields under the concentration of stress, as then does its next successor when in turn exposed to the stress, and so the piece breaks down in detail. But the pliant oak or lead or copper is affected relatively slightly by such notching, and so is the pliant ferrite, as we know it in the low-carbon steels and wrought irons.

Let us apply this to the pearlite series. The 1.20 per cent carbon steel represented by point *E* in Fig. 2 is a relatively rigid, unyielding material, with but little ductility; the pure ferrite represented by *D* is extremely ductile. The former is nearer to glass, the latter to oak or copper in its properties. Hence it is to be expected that a given degree of notching should remove a far greater fraction of the strength of the former than of the latter. Hence it would not be at all surprising were we to find that the strength of the cast iron corresponding to *E* instead of being  $100 - 60 = 40$  per cent of *E*, or 54,000 pounds, should actually have only say 30 per cent of *E*, or say  $135,000 \times 0.30 = 40,500$  pounds per square inch. Following this reasoning, the tensile strength line *GAF* has been drawn. It is clearly conjectural; yet it appears to fit fairly well both the facts, so far as observed, and our hypothesis.

*Tensile Strength of Malleable Castings.*—The high tensile strength of malleable cast iron castings agrees easily with our hypothesis, and with the view that the size of the particles of graphite as well as the quantity of graphite should influence the tensile strength greatly.

Indeed, the fact that these malleable castings have three features, each of which, according to our hypothesis, ought to lead to high tensile strength, goes to mark the great strength of these castings a support to this hypothesis. In other words, the fact that these castings, which according to our hypothesis ought to be strong, actually are very strong, in itself supports the hypothesis.

The three features of the constitution of malleable cast iron to which I refer are the following:



(1) Its total carbon is in general much less than that of common gray cast iron.

(2) In many of the reported analyses the combined carbon is not far from 1.20 per cent.

(3) Its flakes of graphite are extremely fine, whereas those of gray cast iron are very much coarser.

Now, reasons (1) and (2) are not in themselves sufficient to account readily for the high tensile strength of these castings. Hence we refer part of this high strength to the third reason, the fineness of the graphite itself.

*Ductility.* — (1) Qualitatively. But little persuasion is needed to convince one that interspersing flakes of a non-extensible, i.e., non-ductile (though indeed flexible), substance like graphite through the metallic matrix should lessen its ductility. Ductility implies flow and rearrangement of the particles of the metal, which, as the mass is drawn out, should perform the "ladies' chain" figure of the quadrille, a given particle relinquishing its grasp on some of its initial neighbors and instead attaching itself to new intimates. The least consideration of what takes place during elongation beyond the elastic limit gives us this idea; and the beautiful experiments of Ewing and Rosenhain show that this conception is essentially true. Conceive a ductile metallic ball one inch in diameter gradually drawn out into a thin wire one-hundredth of an inch in diameter and 555 feet long. When we begin to elongate the sphere and before the elastic limit is reached, each small particle (or let us say each molecule, whichever conception is the easier) may be supposed to elongate proportionally, like the particles of a rubber ball, each molecule retaining its initial contact with all of its neighbors. Hence the return to the initial shape when the deforming stress is released. But when the elastic limit is passed we are forced to suppose that some at least of the molecules part company with some of those with which they were initially in contact, and attach themselves to new ones. Thus our sphere had a surface of 3.14 square inches; our wire has a surface of 209 square inches, or 66 times as much. Now, were our new and great surface composed exclusively of the initial surface molecules, each so distorted as to offer 66 times as much surface as initially, and were this same distortion to extend through the mass, then on releasing the stress these molecules should all resume their initial dimensions, and so

should the mass as a whole return to its initial spherical shape; in short, the elastic limit should not have been passed. The very fact that it has been passed indicates that the individual molecules have undergone something more than the deformation of those of a rubber ball; and this "something more" is, I take it, a regrouping, a ladies' chain, a divorce and remarriage.

To bring about this supposed 66-fold increase of the surface, many molecules which were initially submerged below the surface must have emerged to the surface. Conceiving for the moment that each molecule is a perfect cube, and that all our cubes are laid square, it follows that every submerged molecule has contact with a neighbor on six of its faces, while every emerged one is in contact with only five of its neighbors: in other words, that that one of its six faces which forms part of the surface of the sphere is without such contact. And no matter what shape we assign to the molecules, a like conception forms in our minds. Now the 66-fold increase in the area of surface, i.e., in the number of surface molecules, implies that each of an enormous number of molecules which were initially submerged and in contact with six neighbors, in emerging and reaching the present state of contact with only five neighbors, must at the very least have parted company with one of its initial neighbors; and we cannot resist the inference that this parting has been simply one of many steps, of many linkings and unlinkings, probably thousands of them, during the drawing out from the initial state of a sphere into the final state of a wire.

Our conception that, while distortion within the elastic limit does not imply regrouping of the particles, elongation beyond that limit does, is strengthened by considering the distortion of cotton. The loose wool of cotton\* may be pulled by the hand into every kind of shape, and it is practically wholly inelastic; it does not return to its initial shape. This is because its fibers are so loosely interhooked that they readily give up their old loves for new. As ductility is one manifestation of the power of being elongated beyond the elastic limit, a lump of such cotton

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\* By the "wool of cotton," often called "cotton wool," I refer to the loose cotton before spinning, as distinguished from cotton thread and cotton cloth. It is similar to "cotton batting" and is familiar to all as the soft fluffy, pink, woolly substance which jewelers put loosely into small boxes to hold small pieces of jewelry.



wool may be regarded as extremely ductile in the sense that, while wholly inelastic, it can be distorted or drawn out in any direction and to a very great degree, and that when thus drawn out it retains its new shape instead of returning elastically to its original shape. Now, we may refer this quasi-ductility to the facility with which the unclosed hooks of the loose cotton release one another and immediately hook into new ones. But let us spin that cotton into thread and thereby lock hook into hook so firmly that this divorce and remarriage is no longer possible, and ductility is nearly destroyed. The thread can undergo very little distortion beyond the elastic limit; and this we may say is because remarriage is impossible, and this in turn because the interlocking is so firm that if divorce occurs it destroys the hooks themselves, without which remarriage cannot occur. This may aid our conception that ductility, i.e., the power of being elongated past the elastic limit, of taking great permanent set, implies power of the particles to divorce and remarry.

Now this remarrying power must of course be interfered with by the presence of any foreign body like graphite. Any given molecule can during distortion readily part from its initial neighbors; but how can it embrace a new one from which it is separated by a layer of graphite, even if that be ever so thin?

How then is slag-bearing wrought iron ductile? It is ductile along the grain, because under distortion parallel with themselves the rods of slag do not interfere with remarriage. But it is brittle across the grain because they do interfere with transverse remarriage. This case of wrought iron, then, rather supports than opposes this idea of the necessarily embrittling effect of graphite.

Admitting, then, that graphite should lessen ductility, and provisionally and for the sake of simplicity that this lessening should be proportional to the percentage of graphite (it is of course also dependent upon its distribution), the ductility line for the cast iron should throughout lie beneath that of the matrix, with regular percentage decrease of the gap from left to right.

(2) Quantitatively. Unfortunately there is little published data as to the ductility of cast iron. To fix our ideas let us assume that the most ductile of our present series, i.e., the left-hand end of the diagram, has an elongation of 2.5 per cent, as against 40 per cent for pure ferrite: then plotting on the regularly-

decreasing-gap plan, we have the ductility curve *RS* for the series of cast irons given in Fig. 2.

The assumption of 2.5 per cent as the elongation of the left-hand member of our series, the cast iron with 4 per cent graphite and combined carbon zero, is simply the best guess which I can make from a general knowledge of the facts.

*Hardness.* — Certainly the presence of graphite should make the cast iron as a whole softer than its matrix, so that the hardness line for the cast iron should throughout lie beneath the hardness line *TU* of the matrix. In default of actual data we may provisionally fix the left-hand end of the line by assuming the hardness of ferrite as 4.5 of Mohr's scale, and that of graphite as 1.5, and further assuming that the hardness of our left-hand cast iron will be proportional to its percentage of ferrite and graphite, or  $(96 \times 4.5 + 4 \times 1.5) \div 100 = 4.38$ . This gives us point *V*; then plotting the remainder of the line on the regularly-decreasing-gap plan as a first extremely rough approximation, we get the hardness curve for the series of cast irons, *VU*, plotted in Fig. 2. That is to say, at the right-hand end of the diagram where graphite is zero, the hardness of the cast iron as a whole should equal the hardness *U* of the matrix simply because the cast iron consists solely of that matrix: hence the hardness of this right-hand cast iron should be *U*.

Next, if we assume that the softening influence of the graphite is directly proportional to the percentage of graphite, then the gap between the lines *TU* the assumed hardness of the matrix, and *VU* the hardness of the cast iron as a whole which we are seeking, should decrease regularly as we pass from left to right. That is to say, the gap between the two lines should be a regularly decreasing percentage of the ordinate of *TU*. Hence, since *TU* has been assumed to be straight, *VU* also should be straight.

*How do these Inferred Lines Agree with the Facts?* — The lines for the tensile strength, ductility and hardness of cast iron as a whole which we have now drawn in Fig. 2 are those which are indicated by our hypothesis, the hypothesis that the properties of cast iron are due in very important part to its consisting of a mechanical mixture or conglomerate of (1) graphite and (2) a matrix of ferrite and cementite, which matrix is itself in each case some one member of the pearlite series. Rather, these



lines are a first approximation to those which our hypothesis calls for; a first approximation only, because we have had to make many assumptions on scanty foundation of evidence, and in making some of these assumptions we have for simplicity ignored important considerations. In view of this, we cannot expect a close agreement between our lines and the facts; all that our hypothesis demands at present is that there shall be no glaring disagreement between these lines and our observed facts.

To bring together the existing data so as to test these lines, these inferences from our hypothesis, would in itself be an extremely serious task; and even if all available data were assembled, they would probably be found extremely defective, so that further great labor would be needed to supply the defects.

But we may here in a general way ask how these lines agree with our general knowledge of the properties of cast iron as influenced by its percentage of combined and of graphitic carbon.

This question each reader must answer for himself. To the writer this agreement seems as good as is to be expected in view of the conditions. It is not to be pretended that there is such an agreement as in and by itself constitutes a firm or even an important support for the hypothesis; all that is claimed is that there is no marked disagreement between these lines and our general knowledge, no disagreement too great to be referred reasonably to the other important variables, such as the influence of phosphorus, sulphur, silicon, etc., the rate of cooling, and the considerations which in this paper have for simplicity been purposely ignored.

A crude but easy test of our hypothesis is to compare the lines in the diagram with the tabular wording arranged above and below the diagram itself, showing the "Name of matrix," "Name of the cast iron," "Strength," "Ductility," "Hardness," etc., and indicating the prominent uses of the various classes of cast iron. It seems to me that the grading, properties, and uses here summarized are (1) wholly in harmony with the teaching of our diagram and hence with our hypothesis, and (2) a fair summarized statement of our knowledge of the facts. In short, it seems to me that, judged by this comparison, there is a fair agreement between our hypothesis and the facts.

*Probable Composition of the Strongest Cast Iron.* — Following out our hypothesis that cast irons are essentially a conglomer-

erate of graphite with a metallic matrix, let us next ask what composition we should expect to give the greatest tensile strength.

In considering Fig. 2, we supposed that, in a series of cast irons all containing 4 per cent of total carbon but varying in the distribution of that carbon, the strongest ought, on our hypothesis, to contain 1.20 per cent of combined carbon. Like reasoning applied to any other series of given total carbon, leads to a like inference that the maximum tensile strength for that series should be reached with 1.20 per cent of combined carbon. Let two examples illustrate this:

Let us try to draw in Fig. 4 a line to represent the tensile strength to be expected on this hypothesis in a series of cast irons all containing 4.50 per cent of total carbon, but with the

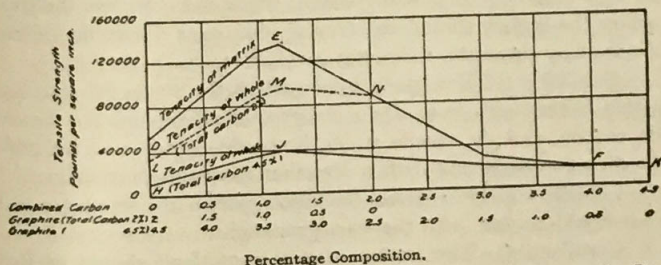


FIG. 4. Expected Influence of Quantity and Distribution of Carbon on the Tensile Strength of Cast Iron containing (HJFK) 4.50 per cent. of Carbon, and of Cast Iron (LMN) containing 2 per cent. of Carbon.

distribution of that carbon varying from graphite 0 combined carbon 4.50 to graphite 4.50 and combined carbon 0.

With graphite 0 and combined carbon 4.50 per cent, the tensile strength curve touches that of the pearlite series at K by the same reasoning which caused that of our 4 per cent carbon series in Fig. 2 to cut it at F. With graphite 4.50 and combined carbon 0, the matrix of our cast iron as before should be pure ferrite, through which should be distributed the flakes representing this 4.50 per cent of graphite. Because this quantity of graphite is larger than that corresponding to point G in Fig. 2, the tensile strength of this present cast iron should be below G; and for like reason the whole curve should be below GAF of Fig. 2, and yet it should be of the family of GAF, reaching its maximum point at 1.20 of carbon. Without quantitative attempt



we may roughly sketch the expected tensile strength curve as *HJFK* in Fig. 4.

In like manner we may draw in this same Fig. 4 the tensile strength curve of a series containing 2 per cent of total carbon. This curve should be somewhat like *LMN* in Fig. 4. For with graphite 0, combined carbon 2 per cent, the line should cut *DEF* at *N*; the left-hand end should be above *G*, because only 2 per cent of graphite is present instead of the 4 per cent which *G* represents; let us set it as roughly midway between *D* and *G*, at *L*. Then, adopting the general family shape of *GAF* and *HJK*, the tensile strength curve for the 2 per cent total-carbon series should be *LMN*, as here drawn.

And so no matter what the total carbon, the tenacity should fall in a curve of this family; and, moreover, the less the total carbon the higher should the tenacity line as a whole lie. From this we may draw the follownig corollaries:

Corollary 1. For given total carbon the strongest cast iron should be that with from 1 to 1.20 per cent of combined or cementite carbon, and the tensile strength should decrease as the combined carbon departs in either direction from this percentage.

Corollary 2. For given combined carbon the strongest cast iron should be that with the least graphite.

Corollary 3. The strongest cast iron of all should be that with 1 to 1.20 per cent of combined carbon and 0.80 per cent of graphite, if we arbitrarily draw the line between steel and cast iron at 2 per cent of total carbon. For on one hand the matrix of such a cast iron would have the composition of greatest strength, and on the other hand, the quantity of graphite by which this matrix is weakened is the smallest which any cast iron with such a matrix can have and yet be a true cast iron, if we follow the arbitrary classification that any pure iron-carbon compound is cast iron if it contains over 2 per cent of total carbon. I purposely say "pure iron-carbon compound," because the boundary between steel and cast iron may be shifted greatly by the presence of elements other than carbon.

Indeed, it is perhaps hardly necessary to go through the sort of crude demonstration here given. The result should follow directly from the hypothesis. The strongest cast iron should be that with the strongest matrix, which we hold corresponds to about 1.20 per cent of carbon, and with the smallest

quantity of the weakening graphite, which by difference is 0.80 per cent. That is to say, we have assumed that nothing can properly be called cast iron which contains less than 2 per cent. of total carbon; this we have taken as a matter of definition. Hence the cast iron which, along with the matrix of maximum strength, 1.20 per cent combined carbon, shall have the minimum quantity of the weakening substance graphite, is that with  $2 - 1.20 = 0.80$  per cent of graphite.

*Test of Corollary 1.* — Of course the tensile strength should be affected by other variables than that here considered, such as the rate of cooling, the size of the particles of graphite, the influence of other elements such as phosphorus and silicon. Hence it is not to be expected that any of these corollaries should be complied with very accurately. But, to get some sort of first crude test, I have plotted in Figs. 5 to 13 all the data which I could find bearing upon corollary 1. For this purpose I have taken all the cases which I could find in which the tensile strength of cast iron, and also its percentage of combined and of graphitic carbon, were given.

From among these many had to be rejected for the reason that there was strong doubt whether the composition given really corresponded to the tensile strength. For instance, in many cases the composition of a pig iron, or unremelted cast iron as it came from the blast-furnace, was given, and along with it the tensile strength of a test specimen made by remelting that pig iron. But this remelting would probably change the composition greatly; so that the tensile strength given would represent a composition not only very far different from that given, but indeterminate. All such data had to be rejected.

I further rejected all cast irons which contained either more than 1 per cent of phosphorus or more than 0.20 per cent of sulphur. This I did because it seemed to me that in all such cases the influence of the proportion of combined and graphitic carbon would be so greatly masked by the variation in these other features of composition that the data would probably be misleading.

After this rejection, I divided the remaining cases according to their total carbon, into eight groups, so that in each group, except the first, Fig. 5, the total carbon varied only by 0.25 per cent. Then, regarding the total carbon of each group as nearly



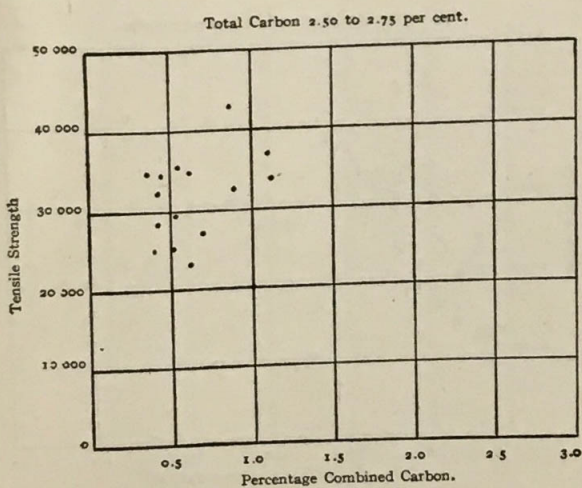
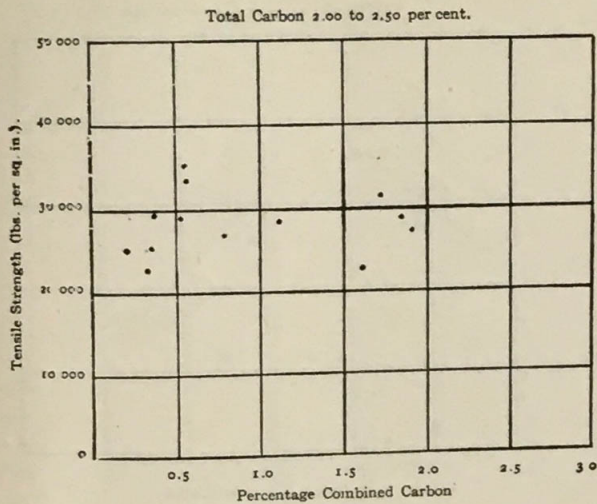
constant, I proceeded to test corollary 1 by plotting the data of each group separately in Figs. 5 to 12, with the tensile strength as ordinate and combined carbon as abscissa. If corollary 1 is correct, then the highest spots in each figure should be in the neighborhood of the 1 per cent or the 1.20 per cent carbon ordinate, and the spots should incline downward to right and left of this ordinate.

In those groups which have the largest number of cases there is a fair degree of correspondence with this corollary. It is certainly true that in these groups, Figs. 7 to 11 inclusive, there appears to be a decided rise towards a maximum as the combined carbon increases from 0 to 1 per cent, and a fall as the carbon increases still farther, though the number of cases with more than 1 per cent is so small that this decrease cannot be traced with so much confidence as the increase from 0 to 1 per cent carbon. For the same reason, it is not clear whether the maximum is at 1.20 per cent or 1 per cent; indeed, the data point to 1 per cent rather than to 1.20 per cent of combined carbon as the maximum point.

It is not pretended that the agreement between this corollary and these data is so close as to support the hypothesis very powerfully. Here, as in our other tests, all that could be hoped was that no glaring disagreement between facts and our hypothesis would be found; and it appears to me that on the whole the agreement is much better than was to be expected.

*Test of Corollary 2.* — The data plotted in Figs. 5 to 12 enable us to test corollary 2 in the following way:

Let us confine ourselves to that part of each group in which the combined carbon is below 1 per cent, because this is the thickly settled part, and let us draw through the axis of each group a line, *ab*, to show the average tenacity for each percentage of combined carbon. This line, according to corollary 2, should be the higher the smaller is the percentage of graphite, or in other words, the smaller is the total carbon-content. Such lines have thus been drawn with care, and they have been assembled in Fig. 14, where it will be seen that as a whole they follow this law, thus supporting corollary 2. In like manner a line has been drawn along the upper boundary of each of these groups, and these lines have been assembled in Fig. 15, where, taken as a whole, they obey this law fairly well, thus further supporting corollary 2.





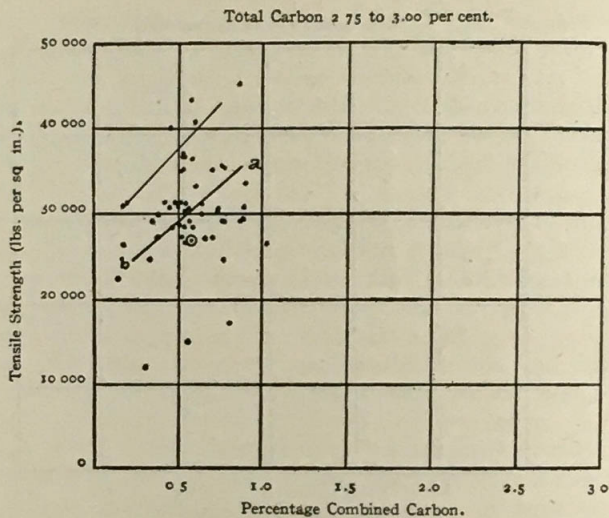


FIG. 7

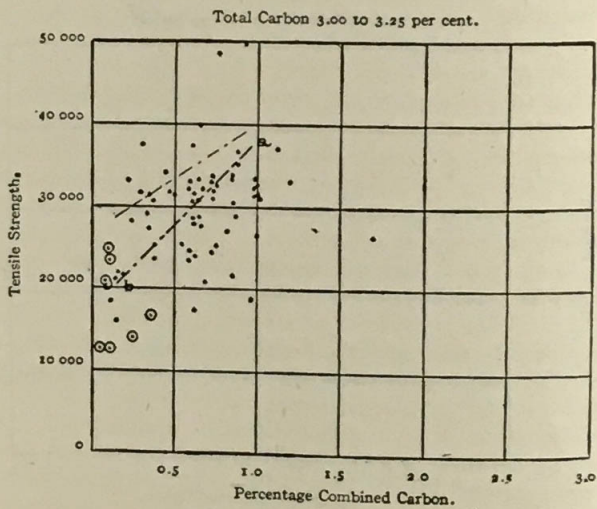


FIG. 8.

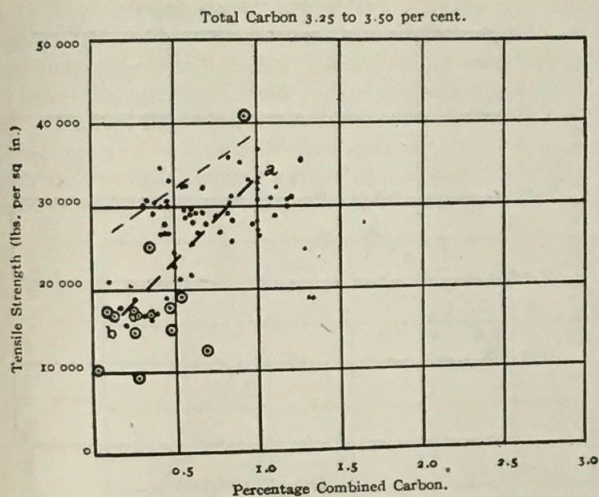


FIG. 9.

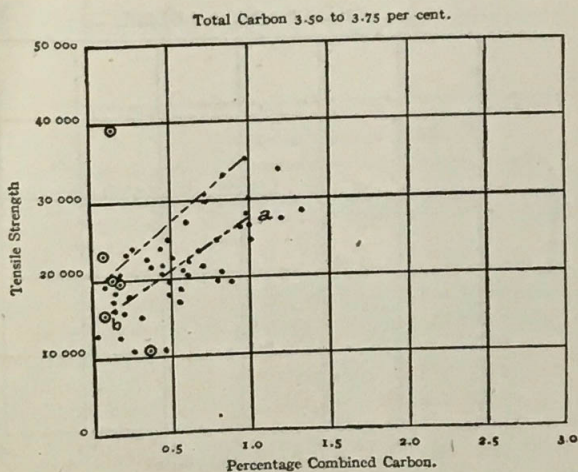


FIG. 10.



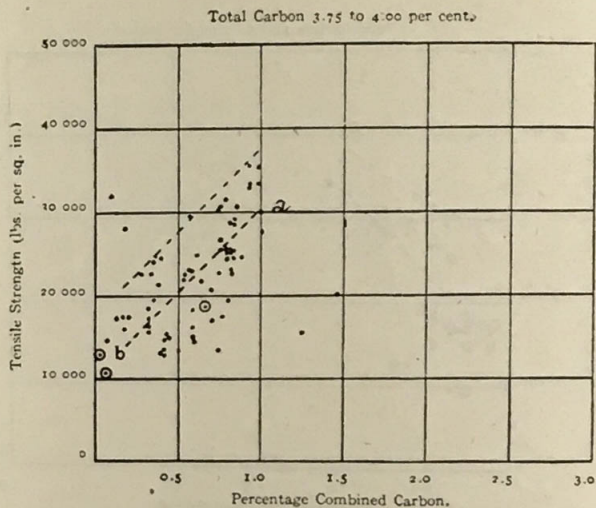


FIG. 11.

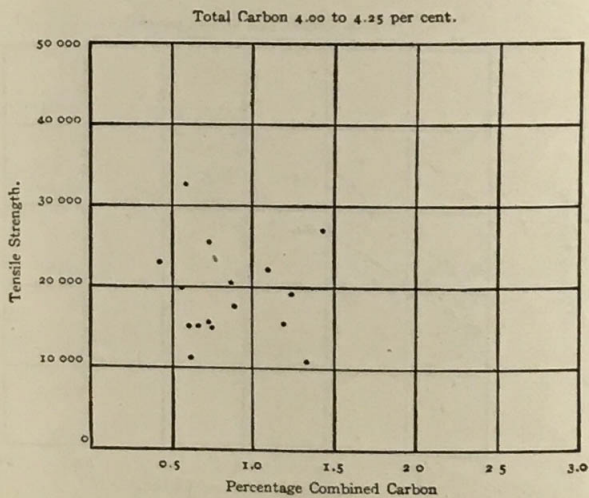


FIG. 12.

In drawing each line in each group, attention was concentrated wholly on this group, so that the lines were drawn without actual knowledge as to how they would compare when assembled.

In examining Figs. 14 and 15 one point should be borne in mind. It is that the ordinate *AB* in each of them passes through or near the center of most of these groups. It gives approximately the average combined carbon of the whole. On this account the relative position of the several diagonal lines at this

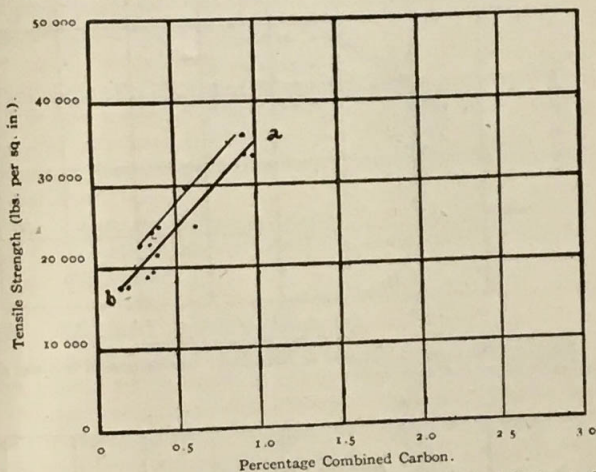


FIG. 13. The Relation between Tensile Strength and the Condition of Carbon in Cast Iron. Series from Data of G. R. Johnson.\*

Silicon, Sulphur, and Phosphorus constant, Combined and Graphitic Carbon variable. (Silicon, 1.20 to 1.29; Sulphur, 0.053 to 0.069; Phosphorus, 0.17 to 0.179; total Carbon, 3.76 to 3.93 per cent.)

\**Four Iron and Steel Inst.*, 1898, II, p. 210, Table I.

ordinate, is particularly important as a test of our hypothesis, and with one exception in each figure these lines follow exactly the order which the hypothesis requires.

The axial and upper boundary lines of Fig. 13 are not reproduced in Figs. 14 and 15 for the sufficient reason that its points have already been included in Fig. 11. These points from Fig. 13 lie higher than the axial line of Fig. 11, and, taken by themselves, rather higher than the hypothesis calls for, i.e., these particular ones taken apart do not agree so closely with the hypoth-



esis as the group, taken as a whole, assembled in Fig. 11 does. The high tensile strength of these irons may be referred to their purity, their freedom from phosphorus and sulphur. On account of this great purity they are not, taken by themselves, strictly comparable with the data in the other figures.

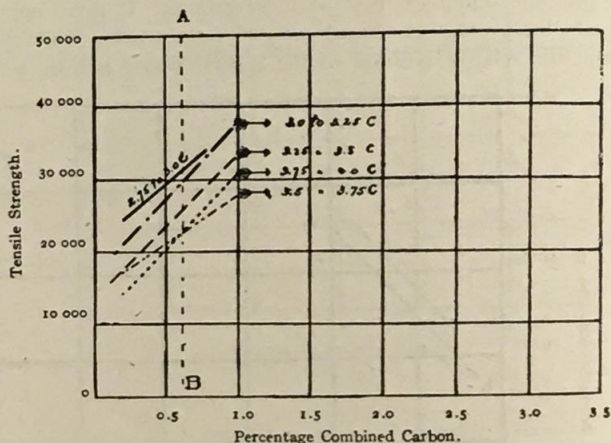


FIG. 14. Axial Lines of Figs. 7 to 11 Assembled. The ordinate A B passes near through the center of all these groups.

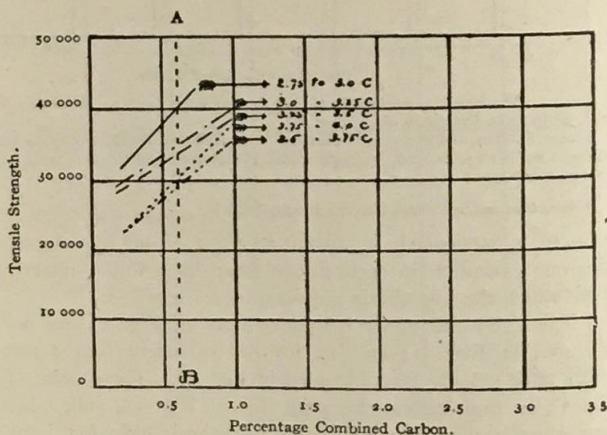


FIG. 15. The Upper Boundary Lines of Figs. 7 to 11 Assembled.

*Estimate of the Quantitative Influence of Graphite on Tensile Strength.* — The assemblage of lines in Fig. 14 enables us to make a rough estimate of the weakening effect of graphite on tensile strength. Comparing any two of these lines, at any given ordinate the percentage of combined carbon is the same for both lines, and hence the constitution of the matrix, and hence in turn its properties, are the same for both lines. The only difference in composition, then, is the percentage of graphite; and therefore we must assume that this difference in graphite-content is the cause of the vertical distance between the lines at such ordinate. In other words, the cast iron of the lower line at such ordinate has more graphite than the cast iron of the upper line at that ordinate, and it is also weaker by the amount represented by the gap between the two lines there; this gap therefore represents the weakening effect of the difference in graphite-content for these two cast irons.

But, comparing different ordinates for these same two lines, the difference in graphite-content at any one ordinate is the same as at any other ordinate.

Hence we infer that the gap between any two lines is due to the difference in graphite-content between those lines.

Let us select the ordinate *AB* as giving the most trustworthy information, because based on the greatest number of cases. At this ordinate the vertical distance between the highest and lowest lines (or rather between the highest line and the crossing of the lowest two lines because these here cross each other) is about 8,750 pounds per square inch. Taking the upper line as representing total carbon 2.87 per cent and the junction of the lowest lines as representing carbon 3.75 per cent, the difference in carbon-content is  $3.75 - 2.87 = 0.88$  per cent. But, as we have already seen, since the combined carbon is the same for both lines here, this difference of 0.88 per cent is really a difference in graphite-content of 0.88 per cent. If this is the real cause of the difference of 8,750 pounds per square inch, then the influence of 1 per cent of graphite within these limits is  $8,750 \div 0.88 =$  say 10,000 pounds per square inch.

*Summary.* — To sum up the results of this investigation, the way in which the properties of cast iron actually vary with variations in the distribution of the carbon between the combined and graphitic states, as given at the bottom of Fig. 2, is in rough



harmony with my hypothesis. Further, two corollaries from my hypothesis, when subjected to test, agree with it as fully as could reasonably be expected.

From these facts I infer that the hypothesis, because of its veridacity and of this preliminary agreement between it and the facts, is worthy of further testing.

*Addendum, January 27, 1903.* — It is only to-day, after the foregoing has been written and most of it has been set up, and after the foregoing engravings have been made, that I have seen what I ought to have seen before, and probably should have seen but for the pressure under which I have been forced to write much of this article in order to keep my promise to have it in time for the present volume. That to which I refer is the fact that the data at hand, on which Figs. 5 to 15 are based, really enable us to test directly the forecast as to the tensile strength of the three series of cast irons containing respectively 2, 4 and 4.50 per cent of total carbon represented by line *GAF* in Fig. 2 and lines *LMN* and *HJK* in Fig. 4. The 4 per cent carbon line *GAF* is susceptible of the most trustworthy test, and this test we will consider first. Then we will consider tests applied to this same line and the 2 per cent and 4.50 per cent carbon lines jointly.

*Test of the 4 per cent Carbon Line, GAF, from Fig. 2.* — The left-hand part of this line, *GA*, is reproduced as *GA* in Fig. 16, and for comparison the axial and upper boundary lines of the group of data nearest in carbon, with carbon 3.75 to 4 per cent, are reproduced from Fig. 11 as *OP* and *KL* respectively. On comparing these lines it appears that, within the limits here shown, the tensile strength curve *GA* which I evolved by a process of reasoning for iron containing 4 per cent of carbon, actually lies between the upper boundary and the axis of the points representing the actual tensile strength of such iron, and nearly parallel with them. In other words, the position of this line as inferred from our hypothesis lies very close to its true position as shown by this accumulation of data.

Indeed, it ought to, as it does, lie above the axial line, because its position was deduced from the tensile strength of carbon steels, which are relatively free from the hurtful and probably weakening elements, phosphorus and sulphur, while the axial line is based on the data of commercial cast irons which doubtless as a whole contained much more of these hurtful elements.

This idea is supported by comparing the Johnson data for pure cast iron, nearly free from phosphorus and sulphur and containing between 3.75 and 4 per cent of carbon. To do this I have reproduced the axial line from Fig. 13 as the line  $QR$  in Fig. 16. It ought to be closely comparable with line  $GA$  as deduced from my hypothesis; and actually it coincides with it almost exactly. Here then my hypothesis receives welcome support.

The tests which have just been described compare the tensile strength line  $GAF$  of Fig. 2 with the lines representing the actual strength of cast irons containing from 3.75 to 4 per cent

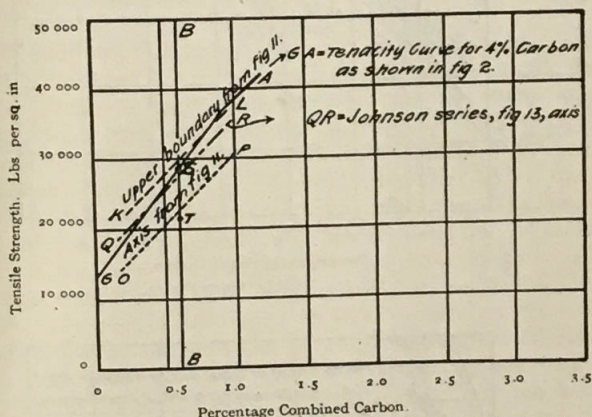


FIG. 16 Comparison of line  $GAF$  from Fig. 2, deduced from my hypothesis, with lines from Figs. 11 and 13, directly-based on experimental data.

of carbon from Figs. 11 and 13. Not content with this comparison, because it is based on a single group of points, that is to say, those within these carbon limits, I wished to compare the position of  $GAF$  with the whole of my data in the following way. I proceeded to calculate at what point the axial line for 4 per cent of carbon, and the upper boundary line for 4 per cent of carbon, should cut the ordinate  $BB$  (given in Figs. 14 and 15 as  $AB$ ) and to see how these points were related to that at which  $GAF$  cuts that ordinate. In making this calculation I took into account the vertical distance, measured at ordinate  $BB$ , between the line for 3.75 to 4 per cent of carbon and each of the other



lines (except that for 3.50 to 3.75 per cent of carbon) in Figs. 14 and 15 respectively. Calculated thus, the upper boundary line should cut *BB* at *S*; and this is actually where *GAF* cuts it: and the axial line should cut it at *T*. In other words, at this ordinate, which represents the thickest dotted part of the field, and

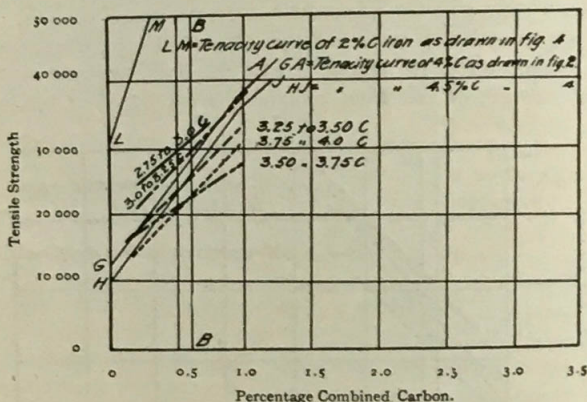


FIG. 17. Axial lines from Fig. 14.

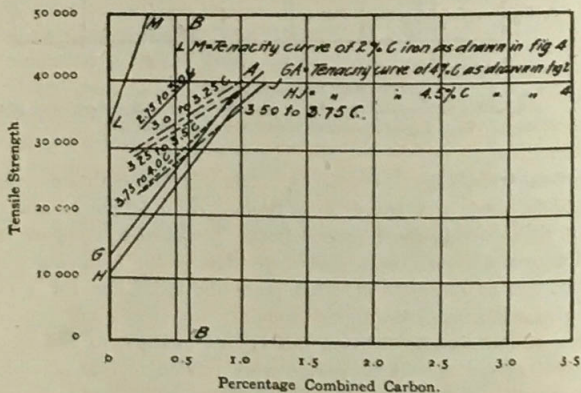


FIG. 18. Upper boundary lines from Fig. 15.

Figs. 17 and 18. Comparison of lines *GAF*, *LMN* and *HJK*, deduced from my hypothesis, with the lines from Figs. 14 and 15, based directly on experimental data.

is therefore the most trustworthy, the calculated strength of cast iron derived from my hypothesis is practically exactly the same as that indicated as the upper boundary by these accumulated data.

*Test of the 2 per cent, 4 per cent and 4.50 per cent Carbon Lines LMN, GAF, and HJK from Figs. 2 and 4.*—I next proceeded to compare these three lines with those of Figs. 14 and 15 by plotting both sets directly from these different figures in Figs. 17 and 18. The parts here plotted of *GA*, *LM* and *HJ* of lines *GAF*, *LMN* and *HJK* may be recognized through bearing here the same letters as in Figs. 2 and 4.

Comparing the position of lines *GA* and *HJ*, for 4 per cent and 4.50 per cent of carbon respectively, with the lines from Figs. 14 and 15, we see that each of the former lies rather higher than it should when compared with the axial lines, and rather lower than it should when compared with the upper boundary lines. In other words, the lines deduced from our hypothesis here too are intermediate between the position which our data accord to the axial and that which they accord to the upper boundary line; so that the position of *GA* and *HJ* compare with the direct data much in the same way that line *GA* does when compared with the lines from Fig. 11 alone. This intermediate position is where we should expect to find them. In short, tested thus, these two tenacity lines as derived from our hypothesis agree reasonably with those based on direct data. But while they are roughly parallel, the former lines are somewhat steeper than the latter, indicating that some additional correction must be introduced into our hypothesis to square its deductions accurately with the facts.

The 2 per cent carbon line *LM*, however, is considerably higher than and much deeper than the data in Figs. 14 and 15 indicate that it should be. This, too, indicates the need of some further correction: though it is to be borne in mind (1) that line *LMN* was not drawn with any attempt at accuracy, but only to show its general shape, (2) that it is so far in composition from what we may call our carefully calculated line *GAF* that it cannot be expected to be accurately placed, and (3) that our data plotted in Figs. 14 and 15 differ widely from it in carbon-content. These facts diminish the weight which should be attached to the discrepancy between the sketched position of *LMN* and the data of Figs. 14 and 15.



**DILATATION OF STEELS AT HIGH TEMPERATURES\***

By GEORGES CHARPY and LOUIS GRENET

WE have undertaken a series of measures of dilatations on quite a large number of samples, including carbon steels and steels with different proportions of nickel. We have employed the dilatometric method of MM. Le Chatelier and Coupeau, which is an application of the method of Poggendorf, by means of a mirror of melted silica for comparing the dilatations of a sample of steel and of a porcelain support, introducing some modifications calculated, as we think, to increase the accuracy of results.

The apparatus for heating is a tube of refractory clay surrounded by a platinum coil, into which an electric current passes. This tube is wrapped in asbestos, varying in thickness at different points, in order to cause an even temperature, and to counteract the cooling influence of the extremities. We have ascertained in this way that three pyrometers, placed in the middle and at the two extremities of the sample of steel, which was 0.06 meter in length, agreed within five or six degrees. The porcelain support does not rest on the heated tube; it passes through the furnace from one side to the other and rests on two outside props.

The support and the piece of steel may also be placed symmetrically with reference to the axis of the furnace, and at an equal distance from the walls; this is necessary for maintaining an even temperature. It was found that the porcelain support was not bent during heating, by substituting a piece of porcelain for the steel. The dilatation of the support was determined with reference to that of quartz parallel to the axis and found equal to  $4 \times 10^{-6}$ , supposing for the quartz an average dilatation of 0.70 per cent between  $15^{\circ}$  and  $570^{\circ}$  C. Finally, we were obliged to place the pyrometer, not by the side of the steel sample, but in a cavity drilled in the steels.

We shall here consider only results concerning annealed steels in the intervals of temperature in which they do not undergo any transformation, intending to take up hereafter the study of the transformation periods and of cold worked or hardened steels.

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\* Comptes Rendus Académie des Sciences, March 3, 1902.

The figures in the following table give result furnished by experiment, without correction, for a certain number of carbon steels.

Composition					Mean Coefficients of Dilatation					
C.	Mn.	Si.	Ph.	S.	15° to 200° C.	200° to 500° C.	500° to 650° C.	24.5x10 <sup>-6</sup>		
0.03	0.01	0.03	0.013	0.023	11.8x10 <sup>-6</sup>	14.3x10 <sup>-6</sup>	17.0x10 <sup>-6</sup>	24.5x10 <sup>-6</sup>	between 880 and 950°C.	
0.25	0.04	0.05	0.010	0.010	11.5	14.5	17.5	23.3	between 800 and 950°C.	
0.64	0.12	0.14	0.009	0.010	12.1	14.1	16.5	23.3	between 720 and 950°C.	
0.93	0.10	0.05	0.005	0.010	11.6	14.9	16.0	2.75	—	—
1.23	0.10	0.08	0.005	0.009	11.9	14.3	16.5	33.8	—	—
1.50	0.04	0.09	0.010	0.010	11.5	14.9	16.5	36.7	—	—
3.30	0.03	0.07	0.005	0.010	11.2	14.2	18.0	33.3	—	—

It will be seen that the coefficients of dilatation, which increase with the rising of the temperature, remain almost equal for the different percentages in carbon up to 650° C. This result agrees with those obtained by Mr. H. Le Chatelier. The fact that nearly pure iron is dilated in the same way as a white casting containing 3.50 per cent of carbon, or nearly 50 per cent of iron carbide  $\text{Fe}_3\text{C}$  (cementite), seems to indicate that iron and iron carbide have essentially the same coefficient of dilatation.

Above the transformation zone coefficients of dilatation almost equal for steels containing less than 0.85 of carbon, are found; above this percentage the coefficients are much higher, but it is possible that the conversions do not terminate completely until a much higher temperature than 700° C. is reached, and consequently influence these numerical values.

Composition of Steels.			Coefficients, averages of dilatation between:				
Ni.	C.	Mn.	15° and 100° C.	100° and 200° C.	200° and 400° C.	400° and 600° C.	600° and 900° C.
26.9	0.35	0.30	11.0x10 <sup>-6</sup>	18.0x10 <sup>-6</sup>	18.7x10 <sup>-6</sup>	22.0x10 <sup>-6</sup>	23.0x10 <sup>-6</sup>
28.9	0.35	0.36	10.0	21.5	19.0	20.0	22.7
30.1	0.35	0.34	9.5	14.0	19.5	19.0	21.3
34.7	0.36	0.36	2.0	2.5	11.75	19.5	20.7
36.1	0.39	0.39	1.5	1.5	11.75	17.0	20.3
32.8	0.29	0.66	8.0	14.0	18.0	21.5	22.3
35.8	0.31	0.69	2.5	2.5	12.5	18.75	19.8
37.4	0.30	0.69	2.5	1.5	8.5	19.75	18.3
25.4	1.01	0.79	12.5	18.5	19.75	21.0	35.0
29.4	0.99	0.89	11.0	12.5	19.0	20.5	31.7
34.5	0.97	0.84	3.0	3.5	13.0	18.75	26.7



In regard to nickel steels, we shall mention only results obtained for a certain number, whose point of conversion is lower than the temperature, and consequently do not undergo conversion in the course of the experiment (reversible steels of Mr. Guillaume).

In this table the dilatations at low temperatures are seen to present the peculiar variations with the percentage in nickel pointed out and studied by Mr. Guillaume; but the coefficients of dilatation increase rapidly with the temperature so as to conceal these variations, in such a way that steels whose dilatations at 100° C. vary in the ratio of 1 to 10 have very similar dilatations at 500° C. and above.

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## TRANSFORMATION OF STEEL BY THE DILATOMETRIC METHOD\*

By GEORGES CHARPY and LOUIS GRENET

THE transformations which iron alloys undergo at a high temperature have been studied especially by the pyrometric method introduced by Mr. Osmond and modified by Mr. Roberts-Austen. This method consists in observing the evolution or absorptions of heat during the heating or cooling of a piece of metal. The study of dilatations enables us to observe the transformation of the metal more closely, to proceed with as low a velocity as desired, even at a stationary temperature, and finally furnishes quantitative indications, while those of the pyrometric method are rather qualitative.

The results obtained by the treatment of steel containing different percentages of carbon and only traces of other bodies, agree qualitatively, whether we operate by heating or cooling, and with different rates of speed. The phenomena of retardation, which assume considerable importance in steels containing such constituents as nickel and manganese, are not, however, negligible in carbon-steels; and in order to obtain precise numerical values, it is preferable to operate by heating and at a sufficiently low

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\* Comptes Rendus Académie des Sciences, March 10, 1902.

speed, so that a diminution in the speed may not modify the temperature of conversion. This result is obtained for carbon-steels by a rate of heating of about 200° C. an hour.

For all steels and cast iron containing only iron and carbon, the conversion begins at about the same temperature nearly 700° C.

At temperatures below 700° C. maintained even for several hours, no sign of conversion is observed.

At 700° C. a sudden contraction appears, the extent of which increases at first with the percentage of carbon, reaches a maximum for steel containing about 0.85 of carbon, and diminishes afterwards when the percentage of carbon tends to increase.

The following table gives the results obtained on several steels:

Percentage of Carbon	Temperature at beginning of contraction Deg. C.	Temperature at end of contraction Deg. C.	Extent of contraction	Critical point A of the pyrometric method
0.03	700	700	hardly	
0.07	680	680	noticeable	
0.15	717	724	1.50	710
0.25	700	710	4.50	712
0.64	703	715	13.0	700
0.93	708	715	13.0	715
1.23	710	713	10.0	715
1.50	694	708	11.0	730
3.50	695	700	9.0	710

When the temperature is gradually raised after this sudden contraction, a more extensive zone of transformation is observed; the dilatation of the metal recommences with a much lower coefficient than before the conversion, a coefficient which rapidly diminishes, when the temperature is elevated, becomes null, and then negative; a new contraction is produced, but it is not sudden, and is distributed over a sudden interval of temperature; then from a temperature varying with the nature of the steel, the dilation recommences with the coefficients indicated.

This second period of transformation extends over an interval of temperature which is the greater as the proportion of carbon is less; it ceases to be noticed distinctly for steels containing more than 0.65 of carbon; for these, only the sudden contraction at 700° C. and above is observed clearly. Only a slight deviation of the



curve of dilatation is produced, of which it is not possible to fix the limits accurately.

The following table gives the limits of this conversion for several soft steels:

Percentage of Carbon	Beginning of the second period	Temperature		Critical points	
		at the maximum of dilatation Deg. C.	End of the second period Deg. C.	of the pyrometric method A <sub>2</sub>	A <sub>3</sub>
0.03	700	860	890	770	890
0.07	680	803	865	770	875
0.15	724	790	840	770	840
0.20	715	740	815	770	825
0.25	710	740	790	770	805

Thus, by the dilatometric method two conversions are observed in the iron carbon alloys: one is abrupt and is produced at 700° C. with contraction of volume which corresponds with the absorption of heat, observed at the critical point A in the pyrometric method; the second, gradual, appears to correspond to a contraction for the steels with less than 0.85 of carbon, and to a dilatation of steels containing more than 0.85 of carbon, and ending at a temperature very nearly that of the critical point A<sub>3</sub> of the pyrometric method. The critical point A<sub>2</sub> which by the pyrometric method is observed at about 750° C. does not correspond to any variation in the phenomena of dilatation.

These results are explained satisfactorily in assuming that the conversions of the iron-carbon systems in the neighborhood of 800° C. resembles those which are produced in the solidification of a mixture of two bodies yielding no mixed crystals. The conversion at 700° C. would correspond to the resolution of the eutectic mixture (pearlite) of iron (ferrite) and of carbide of iron (cementite). The gradual transformation above 700° C. would correspond to the dissolution of ferrite in excess (with contraction) in hypoeutectic steels, and of cementite in excess (with dilatation) in hypereutectic steels.

## EUTECTIC OR BENMUTIC\*

IN your issue of January 24, Professor Howe discusses the convenience of certain new terms in the metallurgy of iron, and I find myself personally drawn into the case. I was the first, or at least among the first, to give the name of eutectic (*acier eutectique*) to the steel which is completely transformed at the lowest atmosphere. As the word "eutectic" signifies, literally, "well melting," and as the question at issue is the designation of a process not accompanied by fusion, I must confess that the criticism of Professor Howe is, from a logical standpoint, incontestably correct.

However, I would plead extenuating circumstances. The formation of languages is not inspired solely by logic, but also by sentiment, and if the word be permissible in matters scientific, by poetry. When, in order to describe a phenomenon which is not fusion, but which in physics is comparable to it, and which follows the same laws, I retain a term which, strictly speaking, is not applicable save to fusion only, assuredly I do so by way of metaphor. But has not this metaphor the advantage of recalling a profound similitude hidden under diverse appearances? The proceeding is usual, and it does not seem to me that it will give rise to any serious difficulty. In all languages, the number of words thus twisted from their first accepted meaning is considerable. For example, we have borrowed from the English language the word "Square," in order to designate the public gardens in cities; it is probable that the first English square was actually square (*carré*), or nearly so, but nowadays we have squares which are round or triangular, and no one bothers about this anomaly of language.

If, however, it should seem desirable to create a new word, I would reproach "benmutic" with being a little too condensed, and I would propose a slight modification, retaining, however, the same derivation. The Latin expression, the adoption of which I

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\* This letter was published in the *Engineering and Mining Journal* for March 28. — For previous correspondence on this subject see *The Metallographist* for April, 1903, p. 167.



would suggest, is "bene mutabilis." "Bene mutabilis" has given us in French *bienmuable* or *benemutable*, according as the word was introduced by spontaneous popular transformation or by the literature of a more recent epoch. As we are talking of an international word, it is the form "benemutable" which best commends itself; it has the defect of being a little longer than "benmutic," but it conforms better to the style of the Neo-Latin languages.

As to the second question, namely, the opportunity of bestowing a name to the solid solution of ferro-carbon and the choice of such a name, I feel no little embarrassment, since it is myself whom my friend, Professor Howe, with a graciousness for which I am profoundly grateful, proposes to choose as sponsor for the term to be created. I am in entire agreement with Professor Howe as to the utility of such a term, especially from the standpoint of instruction. It occurs to me, however, that it would be preferable to wait a bit longer, until our researches into the alloys of iron and carbon are more complete. As a matter of fact, if one turns back to the masterly discussion of Professor Bakhuis Roozeboom ("Zeitschrift für Physikalische Chemie," XXXIV, 437; or "Bulletin de la Société d'Encouragement pour l'Industrie nationale," (5), VI, 609), there is still some hesitation between the diagram 2 and another diagram, such as 5 or 6. If the diagram 2 be correct (and it is the one which appears to me the most probable), the solid solution has forthwith its name. It is "austenite," the only constituent of steel which represents this solution intact, without any beginning of transformation. It seems to me, then, that we could adopt it provisionally. There will always be time enough to introduce a modification if the progress of our knowledge shows the necessity of it; but if it should be confirmed that it is indeed the term "austenite," which is applicable to the case, then Professor Howe would certainly regret, with me, his suggestion of to-day.

F. OSMOND.

Paris, February 24, 1903.

## EUTECTIC OR AEOLIC\*

ACTING on Mr. Brough's suggestion, and with the advice of an eminent Greek scholar, I have decided to adopt the word "aeolic" to designate an alloy of lowest transformation-point as distinguished from "eutectic" or an alloy of lowest melting-point. The two naturally have very much in common; they belong to the same family. The distinction between the two, however, appears to be extremely important, and I do not think that we should be justified by any reasons of expediency in abandoning Guthrie's definition of eutectic. The essential quality of an eutectic, as he defined it, was that it was the alloy of lowest melting-point. On general principles I do not think that we are justified in substituting for this essential and important quality of lowest melting-point, the other and certainly important qualities of the eutectic. It is these latter qualities alone which the eutectic has in common with the aeolic.

It is indeed better to broaden the meaning of "austenite" so as to include what was originally called martensite, and what I proposed to call "osmondite." Indeed, the recommendation of Messrs. Osmond and Sauveur, that this be done, seems to me an admirable one, and I have adopted it.

It is quite true that as M. Osmond says we use the word "square" in speaking of public places, to include also circular and triangular places. To apply this, however, to the present case seems to me to be very misleading. A fair test in all such cases, I think, is this: Is the coexistence of different meanings for the same word liable to cause objectionable confusion? The following illustration will make this clear. We speak of "bow" in the sense of the front of a boat, also as a salutation as when I bow to a friend, also as a particular form of knot as a bow-knot, also as an instrument for projecting arrows, also the act of playing with a violin-bow, etc. Now, these different senses cause no confusion, because they are so distinct that one knows instinctively which sense is meant. So in the case of aeolic which I propose: the fact that this word also has another established meaning is unimportant,

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\* Letter to the Editor received June 5, 1903.



because that meaning is not liable to be confused with the one which I propose.

Now, applying this to the word "square," there is no objection to having this cover circular and triangular public parks, because it is unimportant when speaking of one of these that your hearer should know whether you refer to a true square, a circular, or a triangular park. But when we come to geometry the case is wholly different. If I were to say that every section of a sphere is a square or that every section of a triangular prism is a square, here using the word "square" in the sense of triangle or circle, I should not be understood.

It is exactly so with "eutectic." I say to my students — the eutectic is the alloy of lowest melting-point. Shortly after I say that the iron-carbon compound of lowest melting-point is 4.3 per cent carbon. Soon after I say that eutectic steel contains 0.90 per cent carbon. Now, I happen to know as a matter of fact, that this line of procedure does lead to confusion in the mind of students. I recognize that as a fact proved by my own experience, and on that account and very reluctantly I introduce a new word "aeolic." The truth is that if we regard the whole of iron-carbon compounds as a single series, and that is the point of view important to urge upon students, and if we desire to bring this series into relation with other eutectiferous series, it becomes extremely important that when we speak of the eutectic, we should be correctly understood, and that the students should not be left in doubt as to whether we refer to the alloy of lowest melting-point or to that of the lowest transformation-point.

In what we may for brevity call Roberts-Austen's diagram, i.e., the temperature-carbon-content diagram, of the iron-carbon compounds, there are two distinct sets of curves of the same family, the freezing-point curves and the curves of the transformation from austenite into ferrite and cementite. Each of these sets is of the very first importance, and it is also of the first importance to keep them separate in the student's mind.

This, as a matter of actual experiment, I find is one of the difficulties: the central feature of each set is the alloy of lowest freezing-point or lowest transformation-point, as the case may be; to use the same name "eutectic" for both these is in the highest degree confusing.

It is perfectly true that the parallelism between the two sets

of curves should be made perfectly clear, and impressed on the mind as also of the very first importance; but this the very shape of the curves itself does, as well as all the phenomena of freezing and transformation respectively. To impress this cardinal fact upon the mind, we do not need to take the most objectionable step of calling by the same name, eutectic, these two things which we have difficulty in keeping separate in the student's mind.

In short, to call alloys both lowest melting-point and of lowest transformation-point by the same name "eutectic," quite apart from the objection that it requires complete abandonment of the standard definition of that word, seems to me objectionable because, like using "square" in the sense of "circular" in geometry, it causes confusion; and the fact that such misuse of "square" in case of public parks is harmless, because not confusing, is wholly beside the mark.

The term "saturated" has been suggested for steel of 0.90 per cent carbon. The objection to this is that its implications are wholly untrue. Above the transformation range it appears to take about 1.8 to 2 per cent of carbon to saturate iron. Below that range it appears to take 6.67 per cent of carbon to saturate iron in the sense of converting the whole of it into cementite,  $\text{Fe}_3\text{C}$ . If we were to speak of aeolic steel as "saturated," we might easily be mistaken as referring to one of these two. It appears to me that neither the aeolic ratio nor the eutectic ratio can properly be spoken of as a saturation ratio, and to speak of either of them thus would be in the highest degree misleading.

The objection to specific names, for instance, those ending in "ite," I think, comes from a superficial view. We hear a great deal of adding thus to the terrors of metallography; but I never supposed that the fact that we say "quartz" instead of "hexagonal silica," or "pyrites" instead of "isometric iron bisulphide," or "methane" instead of "carbon tetrahydride," or whatever it might be called, added terrors either to mineralogy or to chemistry. To drop such words as "alcohol," "ether," "phenol," "acetylene," "ethylene," "propyl," and hundreds of others from chemistry and to substitute other words indicating the chemical composition of these various compounds, would be most unwise.

The truth is that the leaders of thought in any branch of science throw new intellectual burdens upon their fellows. The teacher comes along and proposes a new word to help carry this



new burden. He is then abused for increasing the labor of his fellows, but what they really ought to object to is not the conveyance which he furnishes them, but the fact that he is aiding in compelling them to carry that burden. My man has a trunk to carry. I provide him with a truck, and he may tell me that I am making him transport both trunk and truck, but if he has sense he does not say that I am increasing his labor. Among unthinking workmen I may receive (indeed, I have received) a great deal of remonstrance on account of the fact that I introduce wheelbarrows, and compel the workmen to use them in place of trays. Now, it seems to me that this wholesale objection to specific names is of exactly the same order.

It is certainly desirable that our specific names should be systematic. I should welcome any good working system. None exists, none has been proposed. A system based strictly on chemical composition seems to me no more applicable here than in mineralogy; in fact, chemical nomenclature simply swarms with what may properly be called specific names devised to meet the cases in which the fundamental nomenclature, indicating fully the composition or constitution, breaks down through its cumbrousness. It is well that these specific names in turn should be systematic; i.e., that there should be a system in the second degree; but, until a system is proposed, we must, for convenience of language, devise such names as we can, whenever the occasion requires it. If I give you a wheelbarrow, do not scold me because my gift is not the best possible; use it, and be thankful until someone provides a better. The burden of the increasing knowledge is here to be carried; it is my business to force men to carry this load will they nill they; devise for yourselves better wheelbarrows or take the best offered you.

If we look at any branch of physical science, we see that wherever an entity becomes of such importance that it must be referred to often, then somebody proposes a specific name unless a convenient one already exists. In most branches of science this has become so familiar that no objection is raised, but in the new science of metallography many of our friends take a superficial view of the matter, and object to the wheelbarrow for carrying the loads offered.

A wheelbarrow should not be provided for carrying an umbrella, nor should specific names be proposed unless they are to

accomplish some good. Each case then must stand on its merit. A sweeping objection to specific names as such seems to me simply unthinking. A man who has worked much in a particular field may find it necessary to use a specific name which to others will appear wholly unnecessary, for the simple reason that they have not sweated under the load as he has. Therefore, while judgment and discretion should be used in proposing names, I would commend to some of our friends a considerable degree of toleration, for the proposition to create a new name may rest on good reasons which simply have not been made clear to them.

Be it remembered that, when, as in the present state of our knowledge, convenience of language compels us to assign specific names to entities as to whose composition and nature we are in doubt, it is desirable that those specific names should not make implications as to composition or nature, because, if wrong, they later mislead and confuse. The case is wholly different from common chemical nomenclature applied to substances of known composition and, indeed, constitution. Because the case is in its nature like that of mineralogy is the real reason why metallographic nomenclature has followed mineralogical rather than chemical procedure.

HENRY M. HOWE.

New York, June 4, 1903.

*Addendum received June 9, 1903*

P.S. — By *esprit d'escalier* I have contrived, too late to serve me where I sorely needed it, a word which seems to meet the certainly strong objections restricting the "eutectic" to the alloys of lowest freezing-point, while giving me all that I contend for, viz., that the basis of the definition of "eutectic" shall not be completely changed and that we shall be able to distinguish the alloy of lowest transformation-point clearly and easily from that of lowest melting-point. Let us use the word "eutectoid" to denote alloys of lowest transformation-point. The suffix "oid" clearly indicates that the eutectoid has the form or other important properties of the eutectic; it keeps the resemblance of the eutectoid to a eutectic before the mind, while it allows us to preserve the initial meaning for eutectic, and to distinguish between these two really distinct though related entities.



## ABSTRACTS

(From recent articles on Metallography and related subjects.)

**Effect of Strain on the Crystalline Structure of Lead.**

J. C. W. Humphrey. *Philosophical Transactions*, Series A, Vol. 200. — The effects of strain upon the crystalline structure of metals and subsequent effects of annealing at low temperatures have already been studied by Ewing and Rosenhain,\* and the work described in this paper is in many ways a continuation of theirs.

The material used was a pure lead, commercially known as "chemical lead." The first sample obtained had been cut from a casting, and in order to see how the growth of the crystals had proceeded throughout the casting, the rough sides were smoothed with an ordinary wood plane, and the whole casting was subjected to a prolonged etching in dilute nitric acid. The surface of each crystal became covered with a number of geometrical pits, similarly situated over its area, but varying in shape and position from one crystal to another (Fig. 1, natural size). The crystals accordingly showed either bright or dark, as the light striking the sides of the pits was reflected or not into the eye. As the specimen was revolved the crystals flashed out one after another from almost black to brilliant white.

By properly conducted cooling of the molten lead, it was possible to obtain some very large crystals which could be separated from each other and which were used to study the effect of strain upon the internal structure of each grain. These single crystals possessed all the usual plasticity of an ordinary sample of lead made up of numerous small crystals, and could be bent double without breaking. Specimens were cut from these single grains measuring 2 to 3 inches long by  $\frac{1}{2}$  inch wide and  $\frac{1}{8}$  inch thick. These specimens were etched to develop the geometrical pits, and after drying remained bright for a sufficient length of time to enable observations to be made and photographs to be taken. When exposed to the air, the surfaces gradually tarnished. These

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\* *Phil. Trans.* A, Vol. 193, p. 353, and A, Vol. 195, p. 279. Also *The Metallographist*, Vol. III, p. 94, and Vol. V, p. 81.

samples were strained in tension in the small machine described by Ewing and Rosenhain, while being examined under the microscope. As the stress was applied the specimen gradually elongated, this elongation being due to the numerous small slips along the gliding planes of the crystal. These small slips are first visible under the microscope as either bright or dark lines upon the sur-

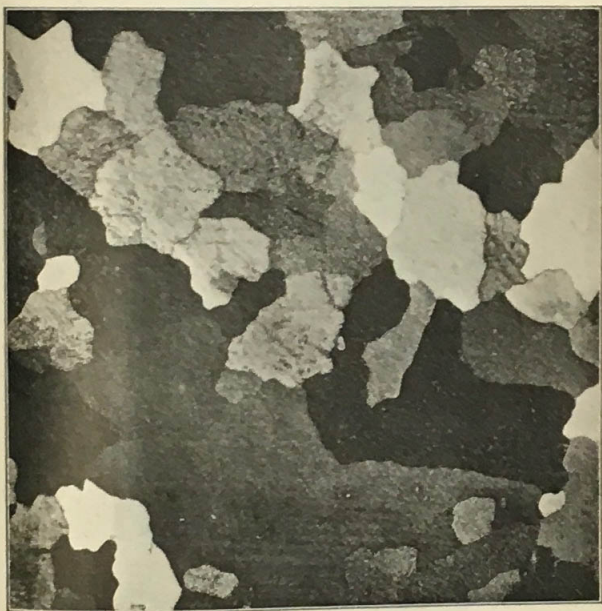


Fig. 1

face, according to the illumination of the specimen, just as Ewing and Rosenhain first observed them; but when the amount of strain becomes large the actual steps formed can be clearly seen. A point of chief interest in the experiments was the relation of the slip-lines to the etched pits upon the surface, that is to say, to the crystalline axes. Fig. 2 is a photograph taken of a surface after straining in tension. The etched pits had been very slowly pro-



duced and are of large size; they are not quite contiguous, but portions of the original surface remain between them. It will be seen that the slip-lines on one face of a pit are parallel to one edge of that face. Again, the illumination is such that a partially formed hexagonal face shows bright, and it will be seen that slip has occurred along planes parallel to this face, so that the small steps also appear bright. From these and other observations it may be concluded that lead tends to slip along planes perpendicular to the octahedral axes of the crystals, and there would, therefore, be at least four possible directions in which slip could occur. Four systems of slip-lines have already been noted in strained lead by Ewing and Rosenhain.

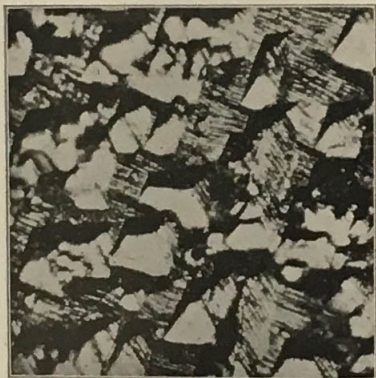


Fig. 2

In these experiments indications were obtained of the production of small crystals by strain, and in order to investigate the phenomenon further, a specimen was carefully cut with a sharp knife from the centre of a single crystal in such a manner that as little strain as possible was given. After etching it was found that the orientation was uniform all through. It was now bent nearly double between the fingers, straightened again, and reetched. Great changes were now visible over the strained area, the orientation no longer being uniform, but broken up into numerous small areas, each with a different orientation. The

greatest change had occurred on what had been the concave side when the specimen was bent, that is to say, where the metal had been subjected to compression. This distortion apparently resulted in the formation of twin crystals, as indicated by numerous straight boundaries between them.

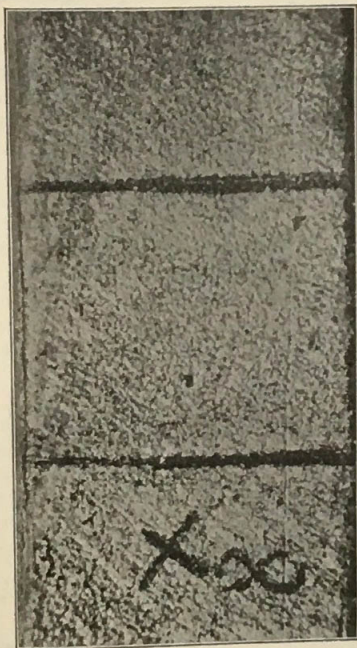


Fig. 3

Ewing and Rosenhain have already drawn attention to the progressive growth of crystals which occurs after a specimen of lead or other metal has been severely strained, especially when the specimen is moderately warmed. Similar experiments were also conducted by the author. A specimen which had been strained by bending was cooked for some hours at a temperature of about



100° C., and then reëtched. After this treatment the area crystallised was far greater than before. That such a growth of new orientations was in some way due to the straining of the specimen was clearly proved by roasting an unstrained specimen. In this no change was produced, but after straining and again cooking the specimen was found upon reëtching to have to a great extent recrystallised. It was also found that it was not necessary for the specimen to show any signs of recrystallisation before annealing, but that so long as a certain amount of strain had been given, whether upon reëtching after this strain the orientation showed any signs of alteration or not, yet after cooking at 100 C. the recrystallisation either continued or was started. Specimens were strained both by bending and tension, and in both cases a further recrystallisation occurred after heating. When a specimen has been strained in tension, only a small area near the fracture shows any signs of alteration when reëtched, but it was found that if such a strained specimen was afterwards heated, the whole orientation could be changed. In studying this effect it was indeed found to be far more convenient to strain the specimens in tension, as the amount of strain to which they were subjected could be more easily regulated.

Fig. 3 shows a specimen after straining in tension and reëtching, magnified to five diameters. The two lines seen running across had been scratched with a sharp steel point, and were originally .1 centim. apart, the straining being carried on until they were 1.5 centim. apart; the other marks were for the sake of identification. The only change visible before annealing was a small amount of recrystallisation along the scratches, where the material was, of course, subjected to fairly severe local strain, and in two places between the lines, where it will be seen the new orientations appear as two small dark patches. Fig. 4 shows the same specimen at the same magnification after cooking for five minutes at 60° C. and reëtching. It will be seen that great changes have taken place; the former patches of new orientation have greatly extended, and others have appeared in various parts of the specimen. The greatest change is at the bottom left-hand corner, where a large area has recrystallised. Fig. 5 shows the same specimen, magnified five diameters after further cooking of twenty minutes' duration at 60° C. The recrystallisation has continued until practically the whole of the original orientation is altered. The

scratches have been almost entirely etched away, and with them the small local changes which they had produced. In cases such as this, where the material had been subjected to a uniform strain throughout, the patches of new orientation go right through the specimen, both sides showing a very similar pattern. In this specimen the

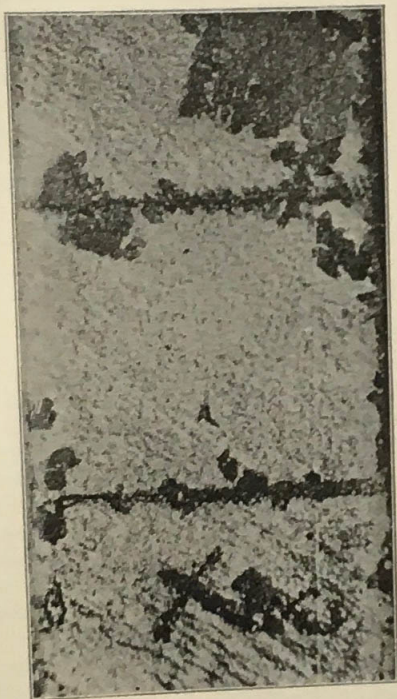


Fig. 4

final formation consisted for the most part of a few large differently oriented areas penetrating right through, each to a large extent split up by twin orientations in bands and patches.

The next experiment was to see whether any change took place in a strained crystal of lead at atmospheric temperatures. A specimen was strained in tension, and stress applied until local con-



traction had commenced, and the strain thus produced was sufficient to cause a certain amount of recrystallisation to be visible immediately upon reëtching. The specimen was kept in a small glass jar, and was simply subjected to the slightly varying temperature of the room. It was found that recrystallisation had



Fig. 5

continued until nearly the whole of one end had changed, the band widening out in both directions so as to fill in the right-hand bottom corner and extend further upwards.

It is clear that, although a further recrystallisation occurs at ordinary atmospheric temperature, yet it is much slower than when the temperature is slightly increased, as had been the case in the

former series. It has been found in all cases that the higher the temperature at which the strained piece is kept the quicker does recrystallisation proceed. In some specimens which had been only slightly strained no visible change was seen until a fairly high temperature was used. One specimen was strained in tension and annealed for twenty hours at  $60^{\circ}\text{C}$ . without any visible change taking place. When, however, it was annealed for two hours at  $100^{\circ}\text{C}$ . recrystallisation occurred.

An important question now presented itself for solution by experiment. Is the recrystallisation which is apparent immediately after etching in a severely-strained crystal a direct and instantaneous effect of the strain, or is it a growth which occurs during the interval of time that has elapsed between the straining and the examination?

From experiments carried on it was inferred that the formation of the patches of new orientation always takes place *after* the stress has been removed and is not directly the result of a general revolution of some of the crystalline elements in the process of straining. From former experiments we have learned that in parts of the crystal where the strain has been severe the patches of new orientation appear almost immediately after the stress is removed and gradually extend from these into the remainder of the strained portion. There appears, therefore, to be no broad distinction between the change which is visible (on re-etching) almost directly after straining and that which takes place after a certain lapse of time. Such differences as are found depend on the amount of strain to which the material is subjected and the temperature at which it is kept; severe straining and a high temperature both tend to increase the subsequent rate of change of structure.

This recrystallisation which has been shown to go on in an individual crystal must be distinguished in one important particular from that which was observed by Ewing and Rosenhain to go on in strained specimens of lead composed of numerous crystals united together by a thin film of eutectic formed of part of the lead united with the metallic impurities. In the present case the specimens were composed of practically pure lead and the action was one of a splitting up of the originally uniformly oriented crystal into numerous differently oriented parts, the action proceeding without the aid of any eutectic. In ordinary lead Ewing



and Rosenhain found that certain crystals gradually increased in size by swallowing up their neighbors, and they have suggested that this was due to a "solution and diffusion of the pure metal constituting the crystals into the fusible and mobile eutectic forming the intercrystalline cement." It is interesting to note in this connection that in the case of a single crystal strained so as to show newly oriented parts, such parts show no inclination to grow into *one another*. When once the whole of the specimen becomes newly oriented, further cooking produces no further change. This, so far as it goes, may be regarded as in agreement with the theory of diffusion through the eutectic, as there would in the case of an originally uniformly oriented crystal be no eutectic between the newly oriented parts, and hence no such growth would be possible.

With regard to the formation of twin crystals we may, however, apply a similar explanation in both cases. To quote from Ewing and Rosenhain's paper: "When a metal solidifies from the liquid state it does so by the formation of skeleton crystals starting from a great number of centres, and the arms of these skeletons continue to grow until arrested by meeting with other growths. From these arms other arms again shoot out, and so on until the entire metal is solidified; but each crystalline element as it settles into place on any of these arms must assume the proper orientation to enable it to fit in, and in the process of filling space by means of such a system of many meeting and interlacing arms the formation of a twin would be almost impossible. But when the metal crystallises after severe strain it does so by the growth of skeleton arms that must often start from a cleavage plane of an actual solid crystal, and probably the new elements deposited upon such a plane would find it as easy to assume the twin orientation as the normal."

In the present case it is exceedingly probable that practically all the patches of new orientation start from a cleavage plane, and hence the formation of twin crystals would be exceedingly common, as in fact it is.

**The Overheating of Mild Steel.** — E. Heyn. — *Journal Iron and Steel Institute*, 1902, II. — The author describes some extensive experiments conducted to ascertain the effect of overheating upon the properties of mild steel. — His conclusions are as follows:

1. When low-carbon mild steel is annealed at temperatures above  $1000^{\circ}$  C. there occurs an increase in the degree of brittleness if the annealing period is sufficiently long. This increase is more considerable and manifests itself the sooner the higher the temperature of annealing. By judicious adjustment of the annealing temperature and annealing period it is possible to produce any desired degree of variation in the brittleness of low-carbon mild steel within definite limits.

2. Prolonged annealing, say uninterrupted for fourteen days, at temperatures between  $700^{\circ}$  and  $890^{\circ}$ , produces no increase in the brittleness. In such cases where the brittleness of the material in its initial state was not yet at the lowest degree possible, by this treatment the lowest degree of brittleness will be attained.

3. Between  $1100^{\circ}$  and  $900^{\circ}$  there exists a temperature limit, above which, if annealing is carried on for a longer period and at an increasing temperature, the degree of brittleness increases. Below this limit, however, this is not the case.

4. Overheating does not only occur at most extreme white heat, but manifests itself already at considerably lower temperatures, which must, however, exceed the temperature limit referred to in (3), the degree being more marked the longer the annealing period.

5. By suitable annealing, the brittleness of overheated low-carbon mild steel can be eliminated. If annealing is carried out at above  $900^{\circ}$  C., a short period of about half-an-hour is sufficient. Longer annealing must be the more carefully avoided, the more the temperature limit referred to in (3) is exceeded, otherwise the signs of overheating reappear. Below  $800^{\circ}$  an annealing of even five hours is not sufficient to eliminate the brittleness in overheated low-carbon mild steel, but by annealing of several days' duration, at temperatures between  $700^{\circ}$  and  $850^{\circ}$ , this object can be attained.

6. If low-carbon mild steel, which has been annealed for a longer period at a high enough temperature, so that after undisturbed cooling it would show extreme brittleness, is rolled or forged during cooling to bright red-heat, it will exhibit no brittleness when cold.

7. The fracture of overheated low-carbon mild steel generally shows a coarse grain, although this is not necessarily always the case.



8. The single crystal grains of which the structure of the iron is built up, which can be detected under the microscope by suitable etching, are often of considerable dimensions when in the state of overheating. Nevertheless this is not to be considered as proof positive that overheating has taken place, since the method of cooling also exercises a great influence on the size of the ferrite grains. Rapid cooling from the temperature causing overheating produces fine ferrite grains, without reducing the brittleness appreciably. Moreover it is possible, by heating low-carbon mild steel for days together at between  $700^{\circ}$  and  $890^{\circ}$  C., to bring the material into such a condition that it will show exceedingly coarse ferrite grains, and yet not exhibit brittleness.

**On the Simultaneous Presence of Structurally Free Ferrite and Structurally Free Cementite.**—In connection with the discussion of this subject which appeared in the last number of *The Metallographist*, the following extracts from Professor Howe's new book on alloys will be of interest:

*Reasons for the Spherulitic Structure of Certain Eutectics.*—My own observations lead me to believe that the banded sheet-like structure, with its zebra-like markings, is the normal structure, the structure with which the eutectic habitually comes into existence; and that the spherulitic structure is due to the drawing together or coalescing of the initially distinct particles of one of the two constituents of the eutectic. Indeed, the separate particles of each constituent may coalesce into larger but still distinct masses. Thus I found that in an alloy of 97 per cent of copper with 3 per cent of silver by weight, when cooled very slowly, the eutectic at the first sight seemed to be entirely lacking. There were little white lakes of silver in the great ground mass of copper; and towards the middle of these lakes were a very few small islands of copper. This was exactly what I had expected.

The explanation is extremely simple. The eutectic itself doubtless consisted initially of sheetlets of argentiferous copper and other distinct sheetlets of cupriferous silver. But during the prolonged stay at a temperature very slightly below the freezing-point, the copper particles of the eutectic slowly migrated outwards, so as to coalesce with the continent of copper surrounding the pool of eutectic, and at the same time the silver particles coalesced so as to form a lake instead of a banded mass. The

little islands of copper remaining were simply those which had not yet coalesced with the outer continent.

Again, on heating an alloy of 63 per cent silver and 37 per cent copper for seven hours to a temperature but slightly below the eutectic freezing-point I obtained similar results. This alloy should normally consist of copper as the excess-metal and the eutectic, and under common conditions we should have large islands of copper in what we might call a marsh of eutectic, but in this alloy I found that the eutectic marsh in the neighborhood of the islands of copper has converted itself into a free littoral region, i.e., around the islands of copper there was a free littoral region of silver containing no bands of copper, from which I infer that the bands of copper initially present in this littoral region had coalesced with the islands of copper.

Further, the eutectic proper, while still distinctly recognizable, had lost to a great degree its banded structure, and the sheets of copper were in large part replaced by minute circular islands of copper.

This coalescing and outward travel is like that of the bubbles floating on the surface of water in a tumbler. The bubbles initially near the edge of the tumbler quickly move outwards and attach themselves to the walls of the tumbler. Those initially nearer the centre, with less attraction towards the walls, and with the attraction of one wall partly offset by that of the opposite, move very slowly; but, given time enough, all will reach the sides of the tumbler.

In quite the same way, it seems to me, we can explain the spherulitic structure of certain eutectics. There has been, at a temperature high enough to give much mobility, a sojourn so long that the sheets of one component have drawn together into little spheres, like so much oil in water.

It has sometimes been said that in a eutectiferous alloy "structurally free" particles of the two constituents of the eutectic cannot coexist. This is wholly incorrect. It is true that there cannot be at the same time an excess of both constituents over the eutectic ratio. If, for instance, silver is in excess over the eutectic ratio, copper cannot simultaneously be in excess. But here is no reason why the particles of the eutectic should not so coalesce, each constituent by itself, that each forms masses of considerable size which, to the eye, are structurally free. Let us recognize that



structural freedom may be an accident; as in the case described above, the metal in deficit may form structurally free masses. Hence, for precise thought, it is much better to use the expressions "excess metal" and "deficit metal" than "structurally free metal." If I am right in this, then the banded structure of the eutectic may be looked upon simply as an accident, and we have to fall back upon its quality of being an alloy of lowest melting-point and also that it is composed of distinct constituents as its two essential qualities. Of these the former evidently is the distinguishing quality.

*Influence of the Structure of the Eutectic.* — I have often been puzzled by the very great degree of ductility which I have given to certain steels by extremely slow cooling. Now this change in the structure of the eutectic is certainly a possible cause, and one which should be investigated. Steel, as we shall see, is a eutectiferous alloy, and the two constituents of its eutectic are sheets of glass-hard and brittle cementite,  $\text{Fe}_3\text{C}$ , and other sheets of soft copper-like ferrite or free iron. Now it may be well that the prolonged sojourn at a high temperature which extreme slow cooling implies, may enable the sheets of cementite to depart somewhat from their initial sheet form and to change towards the spherical form. Further, it is but reasonable to suppose that rounded spheres of cementite should interfere less with the ductility of the whole mass than the same quantity of cementite in the form of sharp-edged sheets, and that even a fractional change from the sheet-like towards the spherical form should have an effect like in kind, though less in degree.

**The Effect of Temperature on the Tensile Strength of Cast Steel.\*** — We present herewith a diagram plotted from the results of a number of tests, made for the purpose of determining the effect of temperature upon the tensile strength of cast steel. Inasmuch as during the past five years the use of cast steel has become general in locomotive construction, it is thought that the results will be of considerable interest.

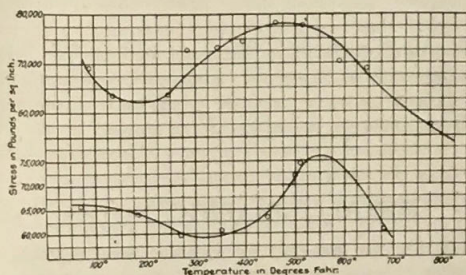
The specimens used in these tests were about 9 inches long and  $\frac{3}{4}$  of an inch in diameter. The specimens after being fastened

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\* From a thesis on "The Physical Properties of Cast Steel," by Messrs. C. C. Schrott and G. H. Case, candidates for the degree of M. E., Sibley College, Cornell University, June, 1902.

in the testing-machine were enclosed in a two-part box. A thermometer was inserted in a cup in one side of the box for taking the temperature. The lower end of the specimen was packed into the box, and the heating substance poured in from above. At first, lard oil was used for temperatures up to  $600^{\circ}$ , but this substance was, in the later tests, displaced by sand previously heated over Bunsen burners.

In the diagram the horizontal scale represents temperature in degrees Fahr., and the vertical scale, stress in pounds per square inch. The two curves represent different grades of steel, and it is interesting to note that in both cases the general characteristics are the same. In the lower diagram the tensile strength gradually



decreases until  $300^{\circ}$  is reached. From this point the strength increases at a greater rate up to  $550^{\circ}$ , where it is a maximum. From this point the strength again decreases as the temperature increases. The upper curve shows a maximum stress of 78,000 lbs. at  $475^{\circ}$ , and the other 76,000 lbs. at  $550^{\circ}$ . Beyond these two points the strength falls off as the temperature continues to rise, and at points from  $700^{\circ}$  to  $775^{\circ}$  the strength is much lower than at the atmospheric temperature of  $70^{\circ}$ . It was also found that the ductility in per cent varies in an opposite way to the strength. Another peculiarity is the character of the break in the test piece. At the lower strength the break is of an ordinary nature, being along a plane perpendicular to the axis of the specimen, but in the higher part of the curve the metal shears in two along a plane at about  $45^{\circ}$  to the axis.



**Etching on Metals by Means of Electric Current.** A. H. Sirks. (Konink. Akad. Wetensch. Amsterdam, Versl. II, 1902.) Abstracted in *Science Abstracts*, Jan. 26, 1903. — The author describes a new method of separating crystals out of any cast metals and alloys, in order to ascertain the properties and composition of those substances. A polished surface of the material to be tested is etched upon by using it as the anode of an electric current, a piece of copper being the kathode. The current was supplied by an accumulator battery of an e. m. f. of 4 volts, and water containing six drops of 10 per cent sulphuric acid to every 100 c.c. served as electrolyte. The author summarizes the advantages of this method as follows: (1) Results may be obtained in cases where the usual polishing and etching method fails; (2) the preparations obtained will show much more detailed drawings and stronger relief than usual etching-preparations; (3) polishing need not be so careful with as the usual method; (4) crystals or crystal fragments, as separated from different alloys by this method, showed on being analysed remarkable differences with respect to the average percentage of the alloys.

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## METALLOGRAPHIC NOTES

**A New Etching Reagent for Polished Steel Sections.** — Mr. F. N. Speller, chemist for the National Tube Co., suggests the following method of developing the structure of iron and steel specimens: From 2 to 4 c.c. concentrated nitric acid is slowly run into 100 c.c. C. P. glycerine and the solution well mixed.

After polishing and drying the specimen the surface is treated with a drop of C. P. glycerine which is gently rubbed on the steel with the tip of the finger. A drop of the etching solution is now applied and friction with the finger continued until the surface is etched to the degree required.

By fastening the specimen in a suitable holder the progress of the action of the acid may be followed through the microscope and the development of the structure checked at the proper time by wiping the glycerine off with a soft cloth and applying a drop of caustic soda-glycerine for a minute.

Mr. Spelter states that the process works very well with low-carbon steels, the pearlite and granular structure being

sharply defined, while the ferrite remains unstained even after 24 hours' continuous application of the etching solution. The chemical composition of this solution is not positively known, but it probably contains glyceric acid.

It is found desirable to prepare a fresh solution every week and to keep in stock solutions of various strengths. The nitric acid used should not be fuming, otherwise nitroglycerine would be formed, a substance which is very dangerous.

**Metallography at the Technical Schools.** — Mr. Wm. Campbell has received the degree of Ph.D. from Columbia University, his major subject being research work in metallography.

The University of Michigan and the Ohio State University are now fully equipped to carry on metallographic work.

**Recent Publications.** — *Thermodynamique et Chimie*, by P. Duhem; 496 pages; illustrated. Librairie Scientifique A. Hermann, Paris.

The present excursions of students of scientific metallurgy in fields which appear to be very foreign to that art, affords a striking instance of the close relation between all physical sciences. Physics and chemistry are no longer regarded as two distinct sciences with sharply drawn boundary lines between them: chemical physics and physical chemistry are words of current use. The supposed gap between mechanics on the one hand and physics and chemistry on the other has been filled by thermodynamics which firmly bind these sciences together. While to the casual observer it must seem as if a fair knowledge of inorganic chemistry and of the fundamental principles of physics constitute all the scientific equipment required to arrive at an intelligent understanding of metallurgical phenomena, the advanced student of metallurgy, at least of the metallurgy of iron, has discovered that it is not sufficient. When he was led to look more closely than had been done by his predecessors, into the constitution of steel, he reached the conclusion that this important metal was in reality an alloy of iron and carbon, and this discovery increased his interest in alloys in general, and in the modern theory of these substances which was being so brilliantly worked out. Alloys, in turn, were shown to be, contrary to the prevailing belief, actual solutions of the component metals, from which it followed that steel also was a solution of carbon in



iron. The metallurgist now looked into the nature of solutions, into their solubility curves, into the phase rule of Gibbs, this remarkable application of thermodynamics to chemical phenomena, which in the hands of some scientists has recently proved so fruitful. In order to properly understand Gibbs' propositions, however, he must study the fundamental principles of thermodynamics. These principles, the phase rule and its most important applications, the constitution of saline solutions, of alloys, of solid solutions, etc., are treated, in the book we have before us, with great authority and erudition by Professor P. Duhem of the University of Bordeaux, the well known author of several important books.

*Notes on Metallurgical Analysis*, by N. W. Lord; 228 pages; illustrated. Metallurgical Laboratory Ohio State University. This is the second edition, rewritten and greatly enlarged, of Professor Lord's well-known book. It deals in a concise and clear manner with the determination of all the elements likely to be encountered in the ordinary work of a metallurgical laboratory, and includes gas analysis and the testing of fuels. This book should be a valuable guide not only to students but also to those engaged in metallurgical chemistry.

*Les Alliages Métalliques* (Metallic Alloys), by L. Gages; 164 pages. Gauthier-Villars, Paris, 1903. This little book is divided into two parts. In the first part the author deals with the metallurgy and the most important properties of alloys; in the second part with the theory of alloys. Industrial alloys are considered under four heads: (1) Ferrous alloys, (2) Bronzes and Brasses, (3) White or anti-friction alloys and (4) Alloys made up of precious metals and amalgams. The author's study of the modern theory of alloys includes description of the solubility curves of saline solutions, the fusibility curves of binary alloys, the surfaces of fusibility of ternary alloys, the metallography of alloys, the phase rule applied to alloys, etc. The treatment of the subject, while not exhaustive, includes the most recent advances made in our knowledge of metallic alloys. This book should appeal strongly both to the student and the practical man.

*Recherches sur les Aciers au Nickel à Hautes Teneurs* (Nickel Steel Containing High Percentages of Nickel), by M. L. Dumas; 208 pages; illustrated. Vve Ch. Dunod. Paris, 1902. This publication gives us, in book form, the very important contributions of

Mr. Dumas to the Study of Nickel Steel, published in the *Annales des Mines* in April, May and June 1902. It is a treatise on nickel steel which should be studied by all those interested in the properties and applications of this remarkable alloy.

**New Departures in Scientific Research.**—The advances which have been made in knowledge of the internal structure of steel within the past few years by means of the microscope, are patent to all who have had to do largely with this complex material.

The microscopic feature of the "new" metallography, the latter "dealing with the composition, constitution, structure and physical properties of metals and alloys, but not including the art of metallurgy"—as defined in the new nomenclature of metallography—may be said to have had its birth in 1864, but its progress lagged sadly behind its contemporary science, chemistry, in its application to steel—although a number of distinguished investigators were quietly at work upon it—until 1898, when fresh impetus was given to the science in this country by Mr. Albert Sauveur, who established at Boston the quarterly publication, *The Metallographist*, which he devoted to the study of metals, with special reference to their physics and micro-structure, their industrial treatment and applications.

Mr. Sauveur's ability and obviously intense interest in the subject seem to have been potent in early attracting to the new system a score of the foremost scientists of Europe and America—as the list of contributors to the publication attests.

In the short period which has elapsed the new metallography has clearly proved its right not only to recognition but also to high position. Already important modifications in heat treatment in the mills can be traced partly, if not largely, to this influence, the most noticeable, perhaps, being the arrangement put into effect in some of the rail mills whereby steel is held before the final pass until the temperature reaches a point on the descending scale at which it will refine in finishing. There is no reason to doubt that the new system will take position alongside its sister science, chemistry, and that each will supplement the other in advancing to still higher planes the metallurgy of steel.—*Sparks from the Anvil*, Jan. 1903, p. 73.



## CORRESPONDENCE

*Influence of the Rate of Cooling on the Structure of Steel.*

To the Editor of *The Metallographist*.

Sir:

Your article in the April number of *The Metallographist* on the "Influence of the Rate of Cooling upon the Structure of Steel" throws light upon a subject to which I have given considerable thought without being able to draw satisfactory conclusions. Your explanation of dilute pearlite seems to fit the case exactly. We found also that the higher we heated our medium-carbon steel the greater the tensile strength and the smaller the elongation. This was especially true in the cases where we found the network structure. I always make a color-carbon test on the test pieces to guard against possible segregation and find that the carbons check very closely. I add this merely as a suggestion from my own experience in slight reply to your question No. 3 at the close of your article. The following table gives some results of our physical testing:

Temperature to which the Steel was heated	Elastic Limit pounds per square inch	Tensile strength pounds per square inch	Elongation Per Cent in 2 inches	Reduction of area Per Cent
$x$	36,210	62,870	39.5	58.66
$x + y$	54,900	81,200	22.3	21.15
$x + y + z$	46,740	90,220	20.0	26.25

Otis Steel Co.,  
Cleveland, O., April 20, 1903.

ARTHUR W. TAYLOR.

*The Rate of Cooling and the Structure of Steel.*

To the Editor of *The Metallographist*.

Sir:

I read with interest in your issue for April, 1903, the paper which you published jointly with Mr. H. C. Boynton "On the Influence of the Rate of Cooling on the Structure of Steel."

The dark, ill-defined constituent, which you obtained in hastening somewhat from  $1100^{\circ}$  C. the cooling of steel containing 0.52 per cent of carbon, is undoubtedly, as you rightly infer, the constituent which I have called "sorbite." This constituent is produced by regulating the rate of cooling in such a way that while most of the carbon assumes the  $\text{Fe}_3\text{C}$  condition, it has not the time to segregate and form distinctly lamellar pearlite. It is of little importance, at least so far as the definition of sorbite is concerned, that the cooling be hastened from a temperature higher than that of the transformations, or only from that temperature. The fact to which you call attention, namely that the carbon content of sorbite may be considerably lower than the carbon content of pearlite, is nevertheless very important. On the other hand, some sorbite may be produced more highly carburized than pearlite, and I have frequently examined samples of steel containing as much as 1.25 per cent of carbon which did not contain any free cementite. As to the condition of the carbon in sorbite, we lack conclusive evidence. It may be conceived, according to known facts, and as I just said, that the combination of the carbon with iron forming the compound  $\text{Fe}_3\text{C}$  is far advanced. It is not by any means certain, however, that this  $\text{Fe}_3\text{C}$  is identical to cementite. The cooling conditions having prevented the segregation of the carbide, it might be conceived, for instance, that the independent molecules of  $\text{Fe}_3\text{C}$  did not reach their normal condition of condensation or of polymerisation. This suggestion has been made by Professor E. D. Campbell and supported by Baron Jüptner. For my part I think that this conception is still a mere hypothesis, but it is an hypothesis which deserves serious consideration and experimental inquiry.

The first of these two properties — variable carbon content and apparently different condition of the carbon — the existence of which cannot be doubted, suffices alone to characterize sorbite and to justify, leaving aside other reasons already given, the creation of a name the usefulness of which has been and still is questioned by some.

No one more than I admires Sorby's work. Not only has he created Metallography, but he has at once conferred upon it a perfect technology. I never would have attached his name to a constituent which, in my mind, would have been of little importance.

It is true that when distinctions are established in a continuous



series, these definitions are necessarily somewhat arbitrary and ill defined. The distance, however, between fully hardened steel and fully annealed steel is too great not to require special terms, for the corresponding differences in structure, even if their detection under the microscope demands delicate manipulations. These structural differences correspond to important differences in the mechanical properties of the metal. Taking into consideration what is already known concerning the influence of the rate of cooling, and leaving aside hardening proper, it seems to me that it may be said that the investigation of the exact conditions of the genesis of sorbite and of the properties which its presence confers upon steel is one of the most pressing problems of metallurgy.

To derive from such research all possible information, I should like to call attention upon the impact test of nicked test bars. By this method differences between steels treated in various ways are made apparent which are not even suspected by the ordinary tension tests. These tests are especially useful in ascertaining the toughness of the metal, a property which is so closely related to the wearing quality of the metals employed in construction.

Among the many methods which have been proposed to conduct this test, that of Mr. Fremont appears to me especially well adapted to laboratory experiments, because it calls for the use of very small test bars measuring  $30 \times 10 \times 8$  millimeters, and which, therefore, may be readily prepared and treated in various ways. In the case of soft steel these bars are nicked with a saw to a depth of 1 mm., and after placing them upon two knife edges a weight of 10 kilograms is allowed to fall upon the side of the bar opposite to the marked side, from a height of 4 meters. The work done in breaking the bar is recorded by difference and may vary, in the case of soft steel and under the conditions mentioned, from almost nothing to 30 kilogrameters and even more, while the ordinary tension tests would indicate almost identical properties. From the results already obtained, it is certain that this method is of very great usefulness in ascertaining the true effect of any thermal treatment.

F. OSMOND.

Paris, April 22, 1903.

# The Metallographist

A QUARTERLY PUBLICATION DEVOTED TO THE STUDY OF METALS, WITH  
SPECIAL REFERENCE TO THEIR PHYSICS AND MICROSTRUCTURE,  
THEIR INDUSTRIAL TREATMENT AND APPLICATIONS.

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Vol. VI

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No. 4

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## THE IRON AND STEEL METALLURGIST AND METALLOGRAPHIST

IN the first issue of *The Metallographist*, published in January, 1898, the purpose and scope of the new magazine was set forth in an introductory article, in which it was contended that metallurgists, engineers, chemists and scientists in general interested in the new and growing science of metallography found it very difficult to keep informed as to its progress because of the necessity of perusing numerous publications in which articles on metallographic subjects were liable to appear, and one of the aims of *The Metallographist* was to "present to its readers every three months a clear, exhaustive and comprehensive review of what had been accomplished in metallography during the previous quarter."

The favor with which the magazine has been received, its steady growth, the many words of encouragement, have been a source of much gratification to the editor. He has found in these unmistakable marks of appreciation conclusive evidences that his efforts were not sterile, his conduct of the journal not unsuccessful. The editor will perhaps be permitted to reproduce here one of the many unsolicited expressions of appreciation which he has received. Had it been the only one, he would have considered it liberal compensation for his efforts:

"I cannot tell you how much delighted I am with *The Metallographist*. It is a most precious publication. Hardly a day passes that I do not refer



my students to it. I give more references to that, I think, than to all other publications together, and I found in Europe that this admirable work of yours was very highly appreciated. HENRY M. HOWE."

On the part of the editor at least a bond of close association, not to say friendship, has grown between himself and the subscribers to the journal. It is as if they had been working together in perfect harmony, for the last six years, in their desire to promote the advancement and progress of the science in which they were interested. It never seemed to the editor that he alone was conducting this publication. He felt in the preparation of each issue the assistance of his subscribers, and he cannot allow this opportunity to pass without expressing his warm appreciation for their support — nay, coöperation — without which the work could never have been carried on for so many years.

But a few lines will now be needed to explain the important change which has been decided upon by the publishers of the magazine.

During the past years many expressions of regret have been received that *The Metallographist* was not published monthly, and the advisability of this important change has been under careful consideration.

Meanwhile, following a similar line of thought as that which prompted the foundation of *The Metallographist*, it became strikingly evident to the publishers that the metallurgists, engineers and chemists desirous of keeping informed as to the progress of the metallurgy of iron and steel are precisely in the same position as were those persons interested in metallography before the publication of *The Metallographist*. Indeed their difficulty in obtaining the desired information is much greater, because of the much greater breadth of the subject, and of the almost innumerable publications in various languages in which its literature is disseminated. They sought upon this point the advice of many students of the metallurgy of iron, and the one and unanimous opinion received confirmed them in the belief that there was an urgent need for a publication *which would do for those interested in the iron and steel industry what The Metallographist had done so successfully for the students of metallography.*

*The Iron and Steel Metallurgist*, the initial number of which will be issued the first of January, 1904, is the outcome of this consideration. It will be a monthly publication of approximately

one hundred pages of descriptive matter devoted to the iron industry in its various branches, including blast-furnace practice, foundry work, steel making, the treatment of steel, physical and chemical testing, the properties of iron and steel, and statistics. Moreover, the field pertaining to metallography, not only of iron and steel but of other metals and alloys, will be covered thoroughly and exhaustively; and, although *The Metallographist* will cease to exist as an independent quarterly publication, it will, as a part of the new magazine, continue to live, and by reaching a larger field will promote even more than heretofore the progress of the science.

Besides a semi-independent metallographical part, *The Iron and Steel Metallurgist* will include the following sections: (1) Original articles from the pen of our special contributors, among whom are numbered more than twenty of the most eminent metallurgists and engineers of Europe and America, and other authoritative writers; (2) Important articles and papers published elsewhere and reproduced in full; (3) Abstracts from articles and papers of less importance; (4) Metallurgical notes including short items of interest, personal notes, editorial comments, meetings of societies, etc.; (5) Monthly reports from iron and steel centers, both domestic and foreign; (6) Correspondence; (7) Book reviews; and (8) Patent records and statistics.

The publishers appreciate the fact that the value of this magazine to the readers must depend to a large extent upon its exhaustive character. They will never lose sight of this important consideration. It will be their constant endeavor to make the reader feel that through the perusal of *The Iron and Steel Metallurgist* no article of value pertaining to the metallurgy of iron and steel, no happening of importance in the iron world will escape his notice. Notwithstanding their realization of the boldness of the undertaking, and of the magnitude of the subject, the publishers feel confident that they can conduct the journal in a manner that will warrant support and encouragement on the part of those interested in this, the greatest of all Industrial Arts.



## THE METALLOGRAPHY OF NICKEL STEEL \*

By LEON GUILLET

## INTRODUCTION

MR. OSMOND has published several important papers on the microstructure of nickel steel, one of which appeared in the January, 1900, issue of the *Annales des Mines*. From his study of the crystallography of iron, Osmond infers that certain nickel steels being non-magnetic must contain iron in the *Alpha* state and nickel in the *Beta* state.

He studied the microstructure of these steels, and found it to be polyhedric. This is the only paper, to my knowledge, which had been published on the structure of nickel steel when my work was completed. I have since learned, through Messrs. Le Châtelier and Dumas, that another article had been published upon that question by Mr. Osmond in the proceedings of the Society of Civil Engineers of London in 1899. In this article Mr. Osmond is much more explicit, and divides the alloys iron and nickel into three groups, the first similar to ordinary carbon steel, the second with a martensite structure, and the third with a polyhedric structure.

I was led quite unexpectedly to study this question in a very complete manner. I received one day a sample of soft annealed steel containing 12 per cent of nickel, and upon examining it under the microscope, in order to estimate its carbon content, I found that it was made up of nearly pure martensite, which did not disappear upon reheating the steel. Although ignorant of Mr. Osmond's work, I concluded that this phenomenon was due to the presence of nickel.

The Jacob-Holtzer Steel Works sent me three series of nickel steels remarkably pure. One contained 0.120 per cent carbon, the second 0.250 per cent, and the third about 0.800 per cent. In each series the percentage of nickel increased gradually from zero to 30 per cent.

I obtained some steels manufactured at Imphy, and kindly placed at my disposal by the management of the Commentry-

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\* *Bulletin de la Société d'Encouragement*, May 31, 1903.

Fourchambault Co. I was thus able to examine steels containing as much as 92 per cent of nickel, if such alloy may be called steel. For the sake of simplicity and clearness, I shall give my results in the following order:

- (1) Microstructure of cast steels.
- (2) Microstructure of quenched steels.
- (3) Microstructure of re-heated steels.
- (4) Microstructure of cold worked steels.
- (5) Microstructure of steels cooled below atmospheric temperature.
- (6) Cementation and decarburization of nickel steels.
- (7) Research on the regeneration of quenched steels.
- (8) Conclusions.

The following table gives the composition of the various samples used:

#### ANALYSES OF THE NICKEL STEEL SAMPLES

Steels*	Carbon	Nickel	Manganese	Sulphur	Silicon	Phos- phorus
SERIES I. — 0.120 CARBON						
2	0.07	2.23	0.025	0.006	0.070	traces
5	0.125	5.23	0.015	0.004	0.046	"
7	0.125	7.13	0.020	0.005	0.050	"
10	0.132	10.10	traces	0.005	0.100	"
12	0.125	12.07	"	0.002	0.090	"
15	0.110	15.17	"	0.004	0.020	"
20	0.176	20.40	"	0.004	0.025	"
25	0.160	25.85	"	0.007	0.036	"
30	0.120	30.00	"	traces	0.031	"
SERIES II. — 0.250 CARBON						
2	0.206	1.97	0.025	traces	0.030	traces
5	0.198	4.90	0.025	0.003	0.043	"
7	0.225	7.59	0.050	traces	0.081	"
10	0.215	9.79	0.025	"	0.015	"
12	0.223	12.27	0.025	0.002	0.014	"
15	0.225	15.04	traces	0.002	0.052	"
20	0.220	20.01	0.020	0.003	traces	"
25	0.230	25.06	0.020	0.003	0.082	"
30	0.194	27.87	0.025	0.002	0.026	"



Steels*	Carbon	Nickel	Manganese	Sulphur	Silicon	Phosphorous
SERIES III.—0.800 CARBON						
2	0.800	2.20	0.107	0.005	0.100	traces
5	0.776	4.90	0.092	0.004	0.085	"
7	0.815	7.09	0.125	0.003	0.100	"
10	1.05	9.79	0.097	0.004	traces	"
12	0.760	12.27	0.092	0.004	0.086	"
15	0.796	15.04	0.060	0.007	0.091	"
20	0.800	20.01	0.020	0.003	0.089	"
25	0.790	25.06	0.070	0.002	traces	"
30	0.810	29.96	0.030	0.004	0.139	"
SERIES IV.						
92	0.520	92.300	3.730	0.060	0.262	0.030
86	0.385	86.120	1.276	0.018	0.828	0.016
49	0.173	49.04	0.973	0.025	0.105	traces
36	0.183	35.88	0.547	0.042	0.070	"

\* The figures in this column indicate approximately the percentage of nickel in the steels.

### I. MICROSTRUCTURE OF CAST STEEL

Before taking up the microstructure it will be well to recall the results obtained by Mr. Dumas from his mechanical tests and described in his important paper on nickel steel.

From the testing of samples cut from forge bars Mr. Dumas arrived at the following conclusion:

True nickel steels, that is, nickel steels containing no carbon, must be divided into four classes:

- 1st group: Steels containing from 0 to 15 per cent nickel — hard steels. Steels with high elastic limit.
- 2d group: Steels containing from 15 to 21 per cent nickel — very hard steels. Steels with high elastic limit.
- 3d group: Steels containing from 21 to 27 per cent nickel — moderately hard steels. Steels with high elastic limit.
- 4th group: Steels containing over 27 per cent nickel. Steels with low elastic limit.

Let us now look into the results obtained with the three series of nickel steel which I have studied.

*First series.* — Steels containing about 0.120 of carbon. The samples containing from 2½ to 7 per cent of nickel have the same structure as ordinary carbon steel. The pearlite, however, is more

finely divided than in ordinary steel containing the same percentage of carbon. It even appears, although this cannot be

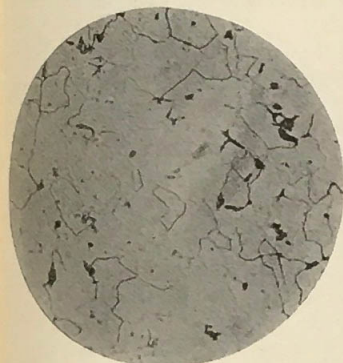


Fig. 1.

Cast steel; 0.120 per cent C, 2 per cent Ni.  
Magnified 300 diameters.

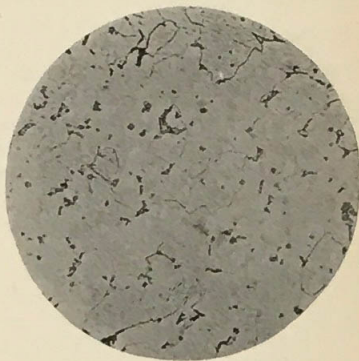


Fig. 2.

Cast steel; 0.120 per cent C, 5 per cent Ni.  
Magnified 300 diameters.

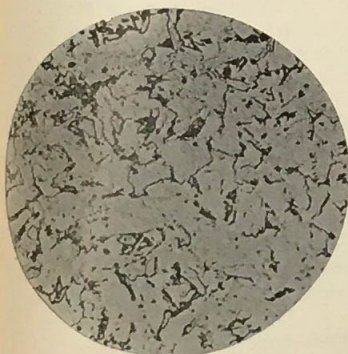


Fig. 3.

Cast steel; 0.120 per cent C, 7 per cent Ni.  
Magnified 300 diameters.

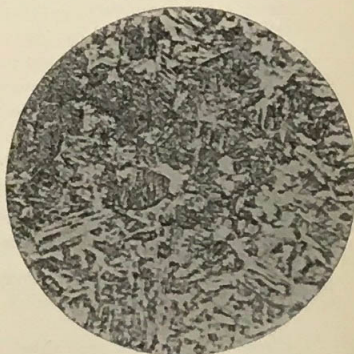


Fig. 4.

Cast steel; 0.120 per cent C, 10 per cent Ni.  
Magnified 300 diameters.

positively confirmed, that the percentage of pearlite increases with the nickel contained (see Figs. 1, 2 and 3).



In the steel containing 10 per cent of nickel we note a change of structure. The martensite structure appears, characterized by its three directions. Some white areas, however, made up of *Alpha* iron, still remain (see Fig. 4).

With 12 per cent of nickel, the white areas have almost entirely disappeared, and the martensite is now nearly pure (Fig. 5). The same is true of steels containing from 15 to 20 per cent of nickel. Steel containing 25 per cent of nickel contains a few white areas which are undoubtedly made up of *Gamma* iron (see Fig. 6). In steel containing 27 per cent of nickel the structure becomes polyhedric (Fig. 7). All the samples were etched with picric acid. All the reagents previously used for the study of the microstructure of steel gave results similar to those obtained in the case of carbon steel, but picric acid is the one which appears to give the best results.

With steel containing over 27 per cent of nickel the polyhedric structure is still obtained, but as the percentage of nickel increases the crystals become, so to speak, more finely divided.

While with from 27 to 30 per cent of nickel the grains are readily colored, when the nickel content exceeds 35 per cent it is no longer possible to color them. As the nickel increases, the time required for the etching also increases very rapidly. With a very large amount of nickel the etching must last several hours. Markings are, moreover, detected in the interior of the grains which appear to indicate cleavage planes.

To sum up, in the case of this first series of experiments, three groups are detected:

- 1st group: 0 to 10 per cent nickel — steels similar to carbon steels.
- 2d group: 12 to 27 per cent nickel — martensite structure.
- 3d group: over 27 per cent nickel — polyhedric structure.

The second group, moreover, must be subdivided as follows:  
From 10 to 12 per cent nickel — steels made up of *Alpha* iron and martensite.

From 12 to 20 per cent nickel — steels made up of nearly pure martensite.

From 20 to 27 per cent nickel — steels made up of martensite and *Gamma* iron.

These results agree with the chemical tests, for it is known that between 12 and 20 per cent nickel the greatest hardness is

obtained, and it will be noticed that between these limits the mechanical properties are very nearly the same.

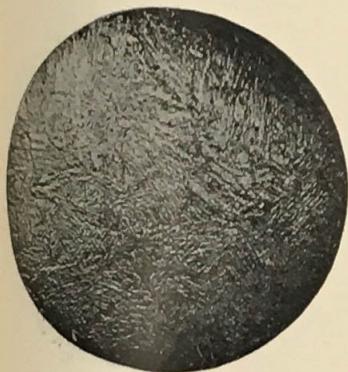


Fig. 5.

Cast steel; 0.120 per cent C, 12 per cent Ni.  
Magnified 500 diameters.

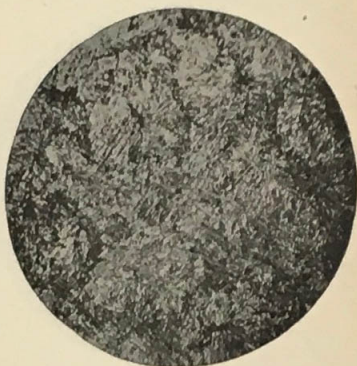


Fig. 6.

Cast steel; 0.120 per cent C, 25 per cent Ni.  
Magnified 300 diameters.

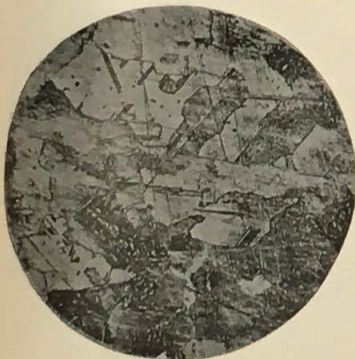


Fig. 7.

Cast steel; 0.120 per cent C, 30 per cent Ni.  
Magnified 300 diameters.

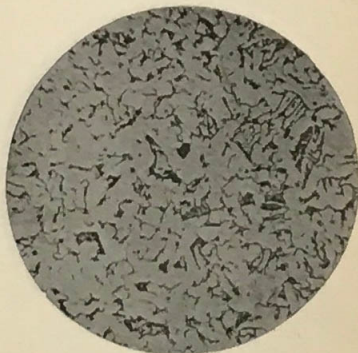


Fig. 8.

Cast steel; 0.250 per cent C, 5 per cent Ni.  
Magnified 300 diameters.

The change of structure with 27 per cent of nickel coincides sharply with the fall of the elastic limit. Finally, I will recall



that the first steel with the polyhedric structure is not magnetic at the ordinary temperature.

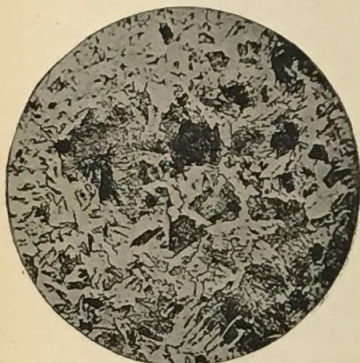


Fig. 9.

Cast steel; 0.250 per cent C, 7 per cent Ni.  
Magnified 300 diameters.

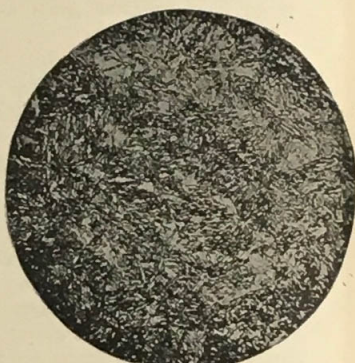


Fig. 10.

Cast steel; 0.250 per cent C, 10 per cent Ni.  
Magnified 300 diameters.

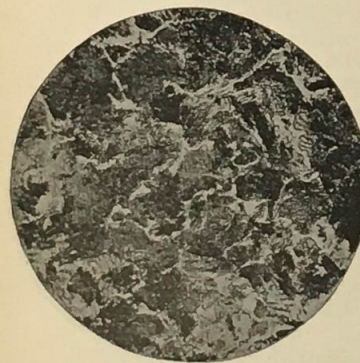


Fig. 11.

Cast steel; 0.250 per cent C, 25 per cent Ni.  
Magnified 300 diameters.



Fig. 12.

Cast steel; 0.800 per cent C, 2 per cent Ni.  
Magnified 300 diameters.

*Second series.* — Steels containing about 0.250 per cent of carbon. From 0 to 7 per cent of nickel, the structure is the same

as that of carbon steel, and my previous remarks concerning the appearance of pearlite appears to be here confirmed (see Fig. 8).

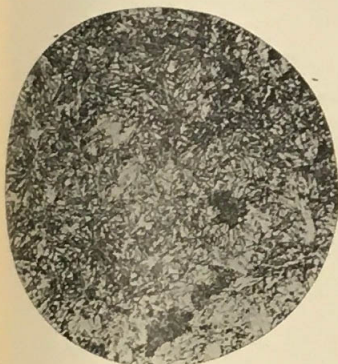


Fig. 13.

Cast steel; 0.800 per cent C, 7 per cent Ni.  
Magnified 300 diameters.

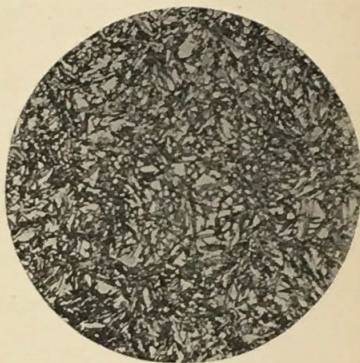


Fig. 14.

Cast steel; 0.800 per cent C, 10 per cent Ni.  
Magnified 300 diameters.

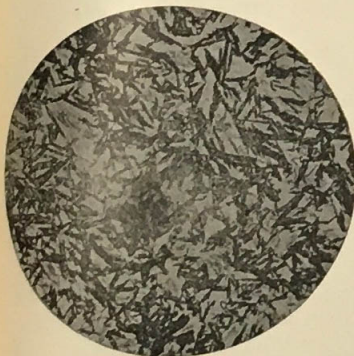


Fig. 15.

Cast steel; 0.800 per cent C, 12 per cent Ni.  
Magnified 300 diameters.



Fig. 16.

Cast steel; 0.800 per cent C, 15 per cent Ni.  
Magnified 300 diameters.

With 7 per cent nickel a little martensite seems to be present (see Fig. 9). With 10 per cent a considerable amount of marten-



site is present (Fig. 10). Between 12 and 15 per cent of nickel martensite is so abundant and so finely divided that it was not possible to obtain a good photomicrograph of the structure. With 20 per cent of nickel large white areas of *Gamma* iron are found.

With 25 per cent of nickel only polyhedric grains are detected, together with a few acicular crystals (*cristals à fer de lance*), the origin of which will soon be explained (see Fig. 11). At about 25 per cent of nickel the same polyhedric structure exists. Here again we have three groups:

1st group: From 0 to 7 per cent nickel — steels similar to carbon steels.

2d group: From 7 to 25 per cent nickel — steels with martensite structure.

3d group: Over 25 per cent nickel — steels with polyhedric structure.

As in the first series, the second group must be subdivided as follows:

From 7 to 10 per cent nickel — steels composed of *Alpha* iron and martensite.

From 10 to 15 per cent nickel — steels composed of pure martensite.

From 15 to 25 per cent nickel — steels composed of martensite and *Gamma* iron.

*Third series.* — *Steels containing about 0.800 per cent carbon.* From 0 to 5 per cent nickel, the same structure is detected, as in the case of carbon steels (Fig. 12).

With 5 per cent, a beginning of transformation is noted. With 7 per cent, martensite is obtained as well as *Alpha* iron (see Fig 13). Pure martensite must occur between 8 and 10 per cent. With 10 per cent of nickel, martensite and *Gamma* iron occur in the structure (see Fig. 14).

With 12 per cent the white areas increase. The same structure is obtained as that resulting from quenching in a freezing mixture at  $-10$  degrees Cent., steel heated to 1050 degrees and containing 1.400 per cent carbon (Fig. 15).

With 15 per cent of nickel polyhedric grains are found, frequently with some acicular crystals, as previously noted (see Fig. 16).

With more than 15 per cent of nickel the structure remains

polyhedric, the crystals, moreover, exhibiting frequently very marked cleavage planes (see Fig. 17).

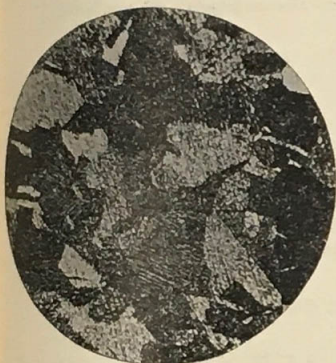


Fig. 17.

Cast steel; 0.800 per cent C, 20 per cent Ni.  
Magnified 300 diameters.

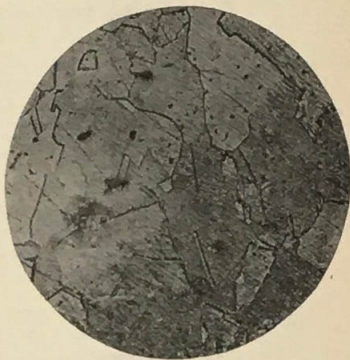


Fig. 18.

Cast steel; 0.175 per cent C, 49 per cent Ni.  
Magnified 300 diameters.

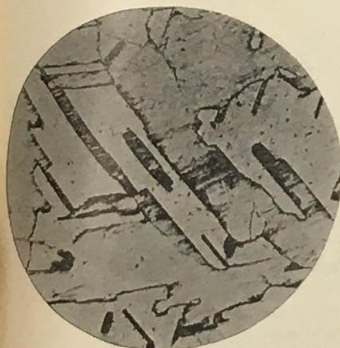


Fig. 19.

Cast steel; 0.520 per cent C, 92.3 per cent Ni.  
Magnified 300 diameters.



Fig. 20.

Hardened steel; 0.250 per cent C, 15 per cent Ni.  
Magnified 300 diameters.

In steels very rich in nickel polyhedric grains are still found (see Figs. 18 and 19).



## CONCLUSIONS

The following table sums up the structural characteristics of nickel steel:

Groups	Micrographic Characteristics	0.12% C.	0.25% C.	0.80% C.
I	<i>Alpha</i> iron and pearlite . .	from 0 to 10% Ni.	from 0 to 7% Ni.	from 0 to 5% Ni.
II	Pure martensite	" 10 to 27 "	" 7 to 25 "	" 5 to 15 "
III	<i>Gamma</i> iron .	over 27 "	over 25 "	over 15 "

The second class must be subdivided as previously indicated.

To sum up the results obtained, the microstructure of cast nickel steel is as follows:

(1) All these steels may be divided into three classes, as shown in the preceding table.

(2) The greater the sum of the nickel and carbon contents the smaller the percentage of nickel at which the structure changes.

(3) In each series, the first steel with a polyhedric structure is non-magnetic.

(4) From the microstructure it is possible to infer approximately the mechanical properties of the steel. It is possible, for instance, to tell whether a sample is hard or very hard, with a low or a high elastic element.

(5) It is even possible to infer the ease with which the steel can be worked. The steels of the third class, for instance, are very difficult to machine, the difficulty increasing with the carbon content.

On the whole, the study of the structure of cast steels confirm the important law given by Mr. Osmond concerning the similarity between the effects of hardening carbon, nickel, manganese, etc.

## II. MICROSTRUCTURE OF QUENCHED NICKEL STEELS

Mr. Dumas has shown that quenching from a light cherry-red heat produces but little hardening of that group of steels which have a high elastic limit, while such treatment greatly softens most of the steels having a low elastic limit. Mr. Dumas

adds that quenching hardens materially, without causing brittleness, those steels whose point of transformation is not far from the ordinary temperature.

Referring to Fig. 20a, showing the transformation points both on heating and on cooling, of nickel steels, it will be seen that the irreversible nickel steels are sharply distinct from the reversible. Mr. Dumas reaches the following conclusions:

From 0 to 21 per cent of nickel, quenching has practically no effect.

In steels containing from 21 to 27 per cent nickel, quenching raises considerably the transformation point both on heating and on cooling.

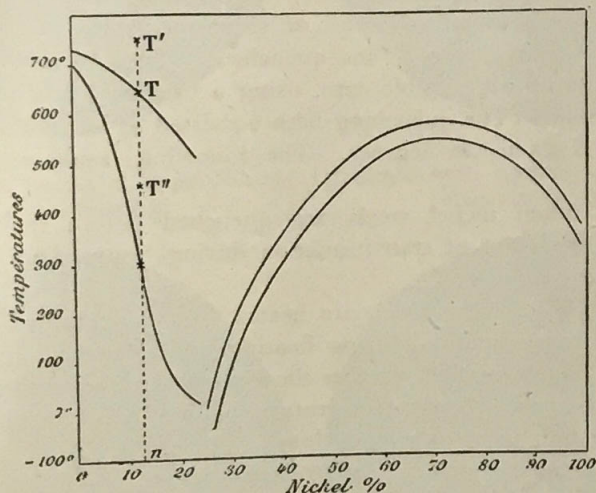


Fig. 20a.

Magnetic critical points of Nickel steel.

From 27 to 29 per cent of nickel, quenching produces a rise of the point of transformation during heating and a very considerable one during cooling.

When the steel contains over 31 per cent of nickel the irreversible transformation can no longer be produced. Quenching slightly lowers the reversible transformations. From the fact that quenching raises the transformation point, it follows that some non-magnetic steel at the ordinary temperature may become magnetic.



In our research we have made the following series of experiments:

In the first series the samples of steel were quenched at a temperature higher than that of the magnetic transformation during heating. A sample of steel, for instance, containing  $n$  per cent of nickel, was in this experiment quenched at a temperature  $T'$  higher than  $T$ .

In the second series the steel was heated to a temperature higher than the point of magnetic transformation during heating, and quenched at a temperature inferior to that transformation. In this case the steel containing  $n$  per cent of nickel was quenched at a temperature  $T''$ , between  $t$  and  $T$  after having been heated to  $T'$ .

Finally, in the third series of experiments the sample was heated to a temperature  $T''$  and quenched. These operations were conducted with all possible care, using a Charpy Furnace and a thermo-couple. The quenching bath consisted of water at a temperature of 15 to 20 degrees. The following results were obtained:

(1) When nickel steels are quenched before they have reached their point of transformation during heating, no change of structure occurs.

(2) Those steels which are heated to a temperature higher than the transformation during heating and quenched, undergo the same transformation whether they have been quenched at that temperature or at a lower temperature, before having reached the transformation point during cooling.

These facts, of course, were to be expected. It only remained for us to ascertain further what takes place when nickel steels are quenched at a temperature higher than that of the transformation point on heating. The results obtained are remarkably clear.

Referring to the groups into which cast steels have been divided, we find that the steels belonging to the first class undergo the same transformation as carbon steels. Martensite and a network of ferrite are formed. The steels of the second group showed a tendency to form a polyhedral structure, this being especially so in the case of the sample containing 0.25 of carbon and 15 per cent of nickel and quenched at 700 degrees. It would seem, therefore, that a part of the iron assumes the *Gamma* state.

Passing now to the steels of the third group, two cases should be considered. Either the point of transformation of

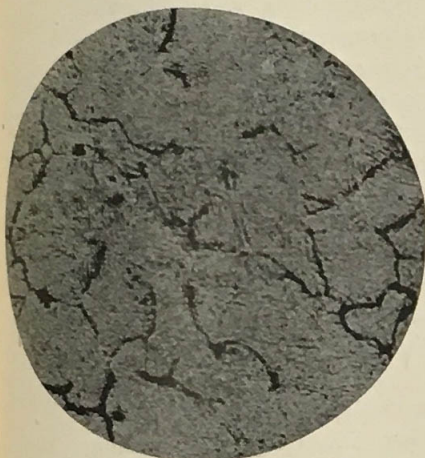


Fig. 21.

Hardened steel; 0.250 per cent C, 25 per cent Ni.  
Magnified 300 diameters.

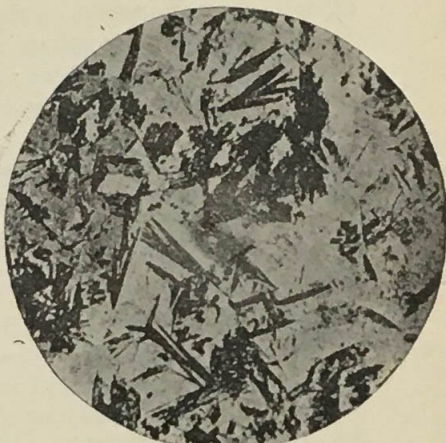


Fig. 22.

Hardened steel; 0.800 per cent C, 15 per cent Ni.  
Magnified 300 diameters.

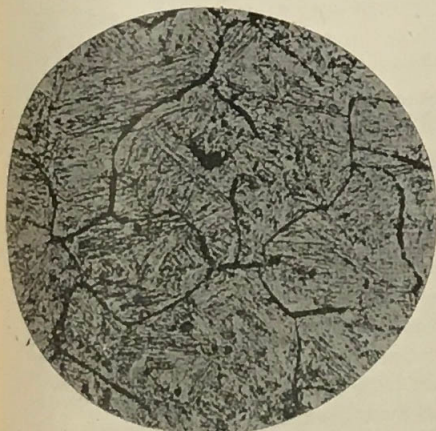


Fig. 23.

Hardened steel; 0.120 per cent C, 25 per cent Ni.  
Magnified 300 diameters.

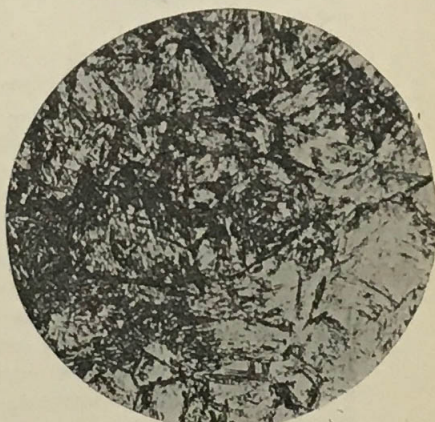


Fig. 24.

Hardened steel; 0.800 per cent C, 10 per cent Ni.  
Magnified 300 diameters.

these steels is raised by quenching or it is not influenced by it. In some cases, as was noted by Mr. Dumas, some steels which



were non-magnetic, or slightly so, at the ordinary temperature became magnetic. We inferred from this that quenching should produce a transformation of the microstructure, and such indeed is the case. Upon quenching at a temperature higher than the transformation point on heating those steels which in each series show first the polyhedric structure, some acicular crystals were formed which recalled those obtained by severe quenching of carbon steels and which are always accompanied by austenite. These steels, however, are white. Figs. 21 and 22 show the structure of steels containing respectively 0.25 per cent carbon with 25 per cent of nickel and 0.8 per cent of carbon and 15 per cent of nickel after quenching. In the last sample some outlines of the polyhedric grains may still be detected. When the steel contains more nickel, quenching has no longer any marked influence upon the point of transformation. The structure remains polyhedric but the crystals appear more finely divided.

In the following table are shown the effects of quenching upon various nickel steels:

Groups	Microstructure before quenching	Effect of quenching.
		Quenching temperature a little higher than transformation point on heating. Quenching bath: Water at 20° C.
I	<i>Alpha</i> iron + pearlite or cementite + pearlite	Same effect as with ordinary steels.
II	Martensite. . . .	Tendency towards polyhedric structure, but predominance of martensite.
III	Pure <i>Gamma</i> iron .	<div style="display: inline-block; vertical-align: middle;"> <math>\left\{ \begin{array}{l} 1. \text{ For the first steels of each series with} \\ \text{polyhedric structure: acicular crystals and} \\ \text{more finely divided crystals.} \\ 2. \text{ No appreciable change in the case of} \\ \text{steel containing more nickel.} \end{array} \right.</math> </div>

It will be seen that these results agree with Mr. Dumas, provided we take into consideration the first group on which the effect of quenching is the same as in the case of carbon steel. Finally, steels exhibiting a martensite structure were quenched under conditions which in the case of carbon steel would produce austenite mixed with hardenite. Polyhedric grains of martensite were obtained. These grains are clearly shown in Fig. 23, while in Fig. 24 it will be seen that the crystals of martensite have a tendency to assume the polyhedric structure. The meaning of the transformation caused by quenching in steels having a polyhedric structure will soon be shown.

## III. MICROSTRUCTURE OF ANNEALED STEELS

Let us first recall that Mr. Dumas has shown that reheating produces results similar to those of quenching. It has a tendency to decrease hysteresis; that is to say, after reheating the distance between the two points of transformation (on heating and cooling) becomes less. This shows that through annealing as well as through quenching these steels which have their points of transformation near the ordinary temperature may from non-magnetic become magnetic.

I have studied the effects of annealing upon the microstructure of nickel steel in a series of experiments. In the first experiments all the samples were heated in an electric heating furnace to a temperature of 900 degrees during four hours. They were then allowed to cool slowly. The results showed that the effect of reheating on the steels of the first group was the same as that resulting from the same treatment of carbon steel.

In the case of the steels of the second and third groups, there was a tendency to form a polyhedric structure similar to that produced by quenching. This might be due to air quenching. Finally the same exceptions should be made for the fifth group as were made in the quenching experiments. The first steels which present a polyhedric structure also contain acicular crystals, but while after quenching these crystals are generally white, after annealing and etching they assume a dark color. I have not been able to ascertain the reason for this difference in color. It will be shown later on that in other instances they occur sometimes black, sometimes white. The effects of annealing are shown in Figs. 25, 26, and 27 which illustrate (1) the structure of steel containing 0.25 and 25 per cent of nickel reheated at 900 degrees during four hours, and (2) steel with 0.8 per cent of carbon and 15 per cent of nickel reheated at 700 degrees. When the steels contain more nickel a tendency to form martensite is detected which was not observed in the quenching experiments. In the case of steel containing 0.8 per cent carbon and 20 per cent of nickel, for instance, the structure was transformed, as shown in Fig. 28. If the metal contains still more nickel, the only structural change consists of an enlargement of the polyhedric grains sometimes enormous, as I shall soon show.

In a second series of experiments it was our purpose to study



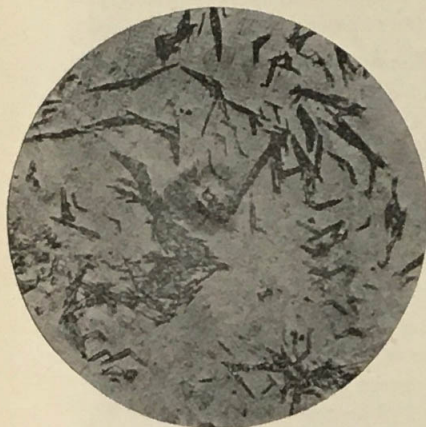


Fig. 25.

Steel annealed at 900°; 0.250 per cent C, 25 per cent Ni. Magnified 300 diameters.

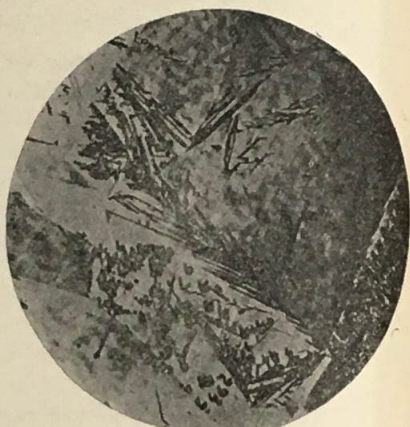


Fig. 26.

Steel annealed at 700°; 0.800 per cent C, 15 per cent Ni. The photograph shows development of polyhedra and formation of acicular crystals. Magnified 100 diameters.

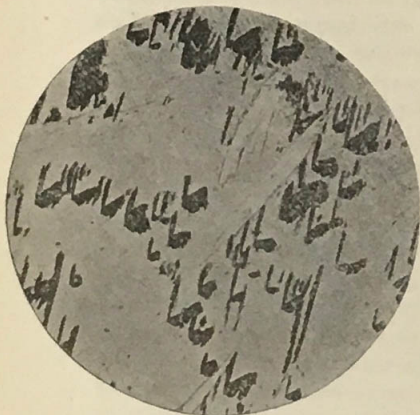


Fig. 27.

Steel annealed at 700° for one hour. The photograph only shows the acicular crystals; 0.800 per cent C, 15 per cent Ni. Magnified 300 diameters.

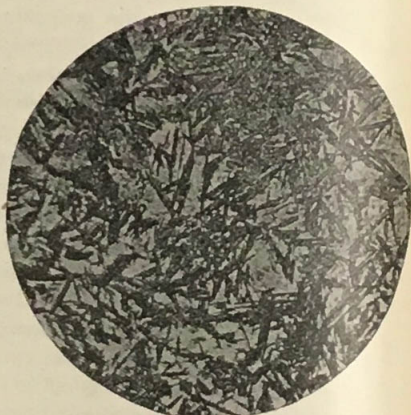


Fig. 28.

Steel annealed; 0.800 per cent C, 20 per cent Ni. Magnified 300 diameters.

the influence of the time during which the steel was reheated upon the microstructure of those samples which form acicular crystals on reheating. For these experiments the samples containing 0.25 per cent of carbon and 25 per cent of nickel and 0.8 per cent of carbon and 15 per cent of nickel were selected. They were heated for different lengths of time, varying between fifteen minutes and four hours, at 900 degrees, and allowed to cool in air. We found that after fifteen minutes there was a decided change of structure due to the formation of acicular crystals. The third series of experiments was then undertaken to ascertain the time required at different temperatures in order to induce the formation of these crystals after cooling. In the case of steel containing 0.8 per cent carbon and 15 per cent of nickel, a decided change of structure was noticed after reheating for thirty minutes at 700 degrees. And this was also the case with the steel containing 0.8 per cent of carbon and 25 per cent of nickel, as shown by Figs. 26 and 27. It would appear as if the time required to produce the transformation does not vary with the carbon content. Some samples were also cooled in the furnace in order to avoid air quenching, but it was found that this air quenching had no influence upon the structure. To sum up, it may be stated that from a micrographic point of view the reheating operation has the same influence upon nickel steel as quenching in the case of all samples except those of the first group. The steels of the first group are affected like carbon steels. Reheating, however, appears to produce a change from the polyhedric structure to a martensite structure in the case of some steels upon which quenching was without effect. We infer from this that when a steel with a polyhedric structure is quenched, resulting in the formation of martensite, the cause of this formation is due to the reheating preceding the quenching, rather than to the quenching proper.

#### IV. MICROSTRUCTURE OF COLD-WORKED STEELS

When nickel steels were introduced it was found that the chips obtained in working non-magnetic steels were magnetic, and that the work could only be continued after quenching or reheating steel. Since then Mr. Dumas has shown that cold-working produces the same effect as quenching. We infer from



this that these steels which have their transformation point near the ordinary temperature must undergo through cold-working important changes in their microstructure. The results obtained are truly remarkable, and the figures only show imperfectly, in spite of the care which was taken, the observed appearances.

For these experiments samples containing 0.25 per cent carbon and 25 per cent of nickel and 0.8 per cent carbon and 15 per cent of nickel were used. According to Mr. Dumas, these samples which were non-magnetic became magnetic through cold-working. He noticed that after cold-working the structure becomes clearly made up of acicular crystals although some polyhedric grains still remained. The most important facts, however, are the following:

(1) The more pronounced the cold-working the more numerous the acicular crystals, and the more magnetic the steel. Careful experiments should be made to confirm this.

(2) The first effect of cold-working is to increase, so to speak, the demarcation of the cleavage planes which are indicated in each polyhedric grain.

A similar effect was noted by Mr. Charpy in the case of brass. It would seem as if this marked the beginning of the transformation of austenite into acicular crystals. This change of structure is shown in Figs. 29, 30 and 31. One of them (Fig. 29) shows some acicular crystals formed on the outside of the polyhedric grains to such an extent that it would seem as if half of the specimen had been hidden by photographing. These photographs appear to throw much light upon the transformations which take place in ascertaining nickel steel and which consist in the change of a polyhedric structure into a structure made up of acicular crystals.

These cleavage planes through their fracture give rise to the formation of acicular crystals. It is difficult to say whether these acicular crystals are identical with the more numerous ones already observed.

To throw more light upon the influence of cold-working, we have made the following three series of experiments, which were by Messrs. Le Chatelier and Mesnager.

In the first series a sample of steel containing 0.8 per cent of carbon and 15 per cent of nickel was cold-worked by means of a press or a hammer without passing the elastic limit. In these

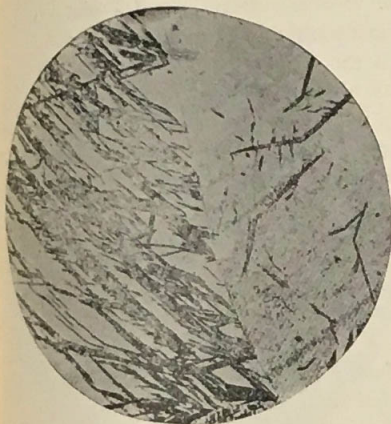


Fig. 29.

Cold-worked steel; 0.800 per cent C, 15 per cent Ni. Magnified 300 diameters. The photograph shows the formation of hardenite near the edge of a polyhedron.

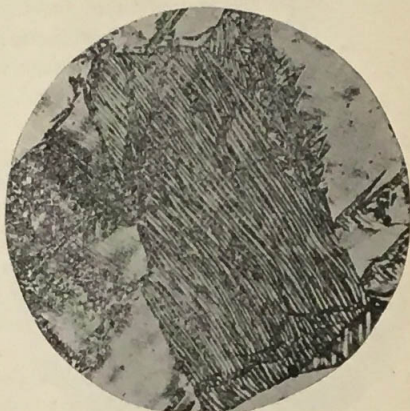


Fig. 30.

Cold-worked steel; 0.800 per cent C, 15 per cent Ni. Magnified 300 diameters. The photograph shows the development of polyhedra and the formation of acicular crystals.

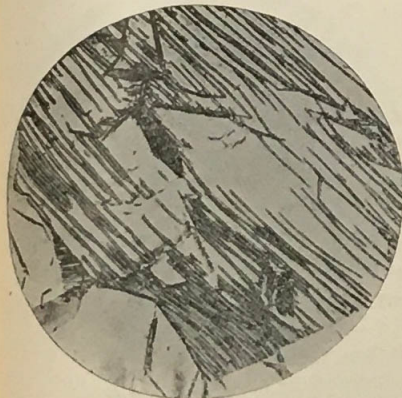


Fig. 31.

Cold-worked steel; 0.800 per cent C, 15 per cent Ni. Magnified 300 diameters. The photograph shows the development of polyhedra and cleavage planes.

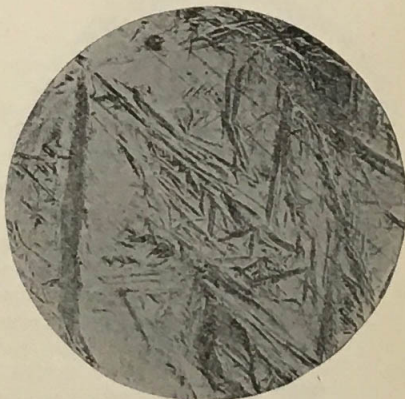


Fig. 32.

Steel cooled to  $-78^{\circ}$ ; 0.800 per cent C, 15 per cent Ni. Magnified 300 diameters. Unetched specimens.



cases the formation of martensite was not observed. The size of the crystals does not increase materially. The surface of the sample in immediate contact with the forging tool, and which had been previously polished, was wholly made up of acicular crystals.

In the second series of tests we passed the elastic limit, and the phenomenon already described was observed, namely, the formation of acicular crystals.

Finally, in another series of tests the elastic limit was passed only in one portion of the mass, and it was only in that portion that the acicular crystals were produced and these crystals increased in number as the surface which received the blow of the hammer was approached.

The development of the polyhedric grains seems abnormal, and it would be interesting to complete these experiments in order to throw more light upon the mechanical effects of work.

It is certain, at any rate, that in the case of those steels whose transformation point is near the ordinary temperature cold-working has a tendency to greatly increase the polyhedric grains and to induce the formation of martensite.

#### V. MICROSTRUCTURE OF NICKEL STEELS COOLED BELOW ATMOSPHERIC TEMPERATURE

According to the work of Dewar, Fleming, Osmond and Dumas, some nickel steels undergo a transformation during the cooling, which is accompanied by a rise of their elastic limit, a great increase of their tenacity and a greater decrease of their ductility. All samples which undergo this transformation, Mr. Dumas states, assume the magnetical characteristics of those steels whose irreversible transformation during cooling is located above atmospheric temperature.

Our first series of experiments dealt with the cooling of various samples to a temperature of  $-78$  degrees obtained by solid carbonic acid and alcohol. It was found that such treatments were without action upon the steels of the four first classes. It had a little effect upon the steels of the fifth class. The tendency is to convert the polyhedric grains into acicular crystals.

The steels containing 0.25 per cent of carbon and 25 per cent of nickel and 0.9 per cent of carbon and 15 per cent of nickel,

which, according to Dumas, become permanently magnetic at  $-78$  degrees, exhibited many acicular crystals. Moreover, under

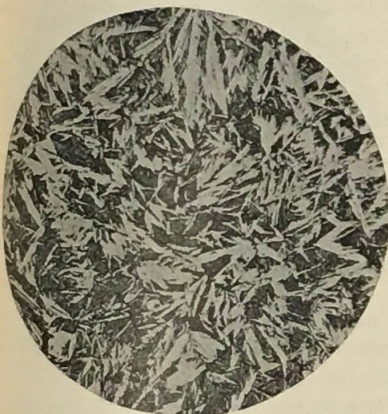


Fig. 33.

Steel cooled to  $-78^{\circ}$ ; 0.250 per cent C, 25 per cent Ni. Magnified 300 diameters.

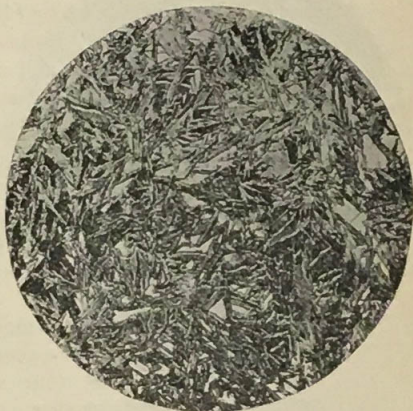


Fig. 34.

Steel cooled to  $-78^{\circ}$ ; 0.800 per cent C, 15 per cent Ni. Magnified 300 diameters.

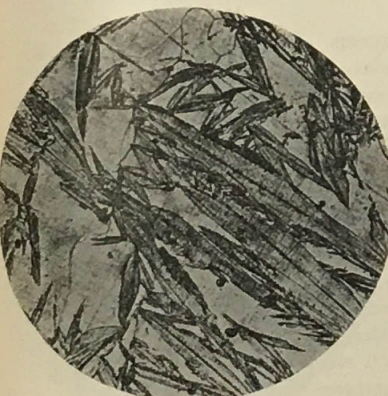


Fig. 35.

Steel cooled to  $-78^{\circ}$ ; 0.250 per cent C, 30 per cent Ni. Magnified 300 diameters.

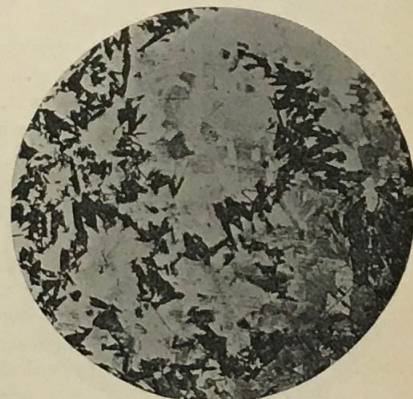


Fig. 36.

Steel cooled to  $-78^{\circ}$ ; 0.800 per cent C, 20 per cent Ni. Magnified 300 diameters.

the influence of cooling the acicular crystals expanded to such a degree that after quenching a surface previously polished the



polyhedric structure could be clearly seen without any need of etching (Fig. 32). This phenomenon has already been observed in the case of quenched steels by Mr. Osmond, who has described it in *The Metallographist* for October, 1899. Figs. 33 and 34 show the same steels after polishing and etching. If the steel contains more nickel than those just mentioned, upon cooling to  $-78$  degrees acicular crystals are formed. Such is the case for steels with the following composition:

0.12 per cent carbon and 20 per cent nickel,

0.25 per cent carbon and 30 per cent nickel,

0.80 per cent carbon and 20 per cent nickel.

Figs. 35 and 36 show the microstructure of the last two of these samples.

With steels containing more nickel no change occurs, but the polyhedric grains show a tendency to increase. The same fact was observed with the four series of steels with which we experimented. The acicular crystals appear sometimes white, sometimes black, after etching. The same sample even exhibits sometimes both white and black crystals. In one instance we found a crystal colored black on one side and white on the other, as shown in Figs. 34 and 37.

In a second series of experiments we have subjected the same steels which had many acicular crystals at a temperature of  $-78$  degrees to coolings to temperatures much below the ordinary temperature. In these steels, even at 0 degree, and after a very short time, a beginning of transformation was observed (Fig. 38). The lower the temperature the more numerous the acicular crystals. At  $-40$  degrees, in the case of the steels experimented upon, they seem as numerous as at  $-78$  degrees (Figs. 39 and 40).

The conclusions of these experiments are as follows:

These steels which are non-magnetic, and which undergo readily the transformation, also undergo a change of structure. Acicular crystals are observed which are generally white. This transformation begins at a temperature near the atmospheric temperature. In the case of some steels which undergo a full transformation at  $-78$  degrees, a change of structure is also noted at that temperature. We have always observed that when acicular crystals begin to be formed magnetism also occurs.

Steels made up of martensite and of pearlite do not undergo

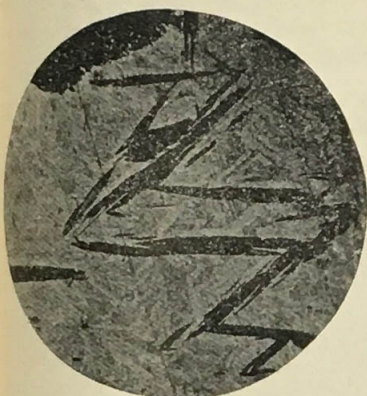


Fig. 37.

Steel cooled to  $-40^{\circ}$ ; 0.800 per cent C, 15 per cent Ni. Magnified 300 diameters. The photograph shows black acicular crystals.

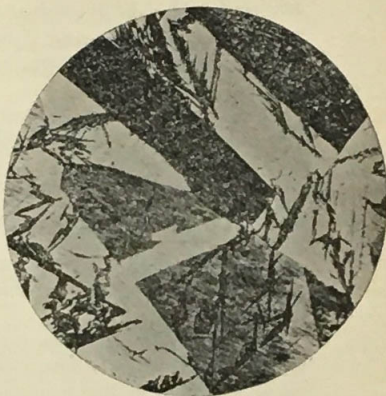


Fig. 38.

Steel cooled to  $0^{\circ}$ ; 0.800 per cent C, 55 per cent Ni. Magnified 300 diameters. The photograph shows the development of the polyhedra, and the formation of acicular crystals.

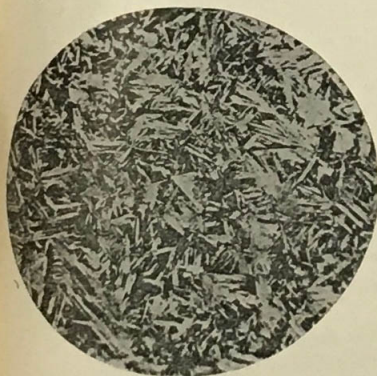


Fig. 39.

Steel cooled to  $-20^{\circ}$ ; 0.800 per cent C, 15 per cent Ni. Magnified 300 diameters.

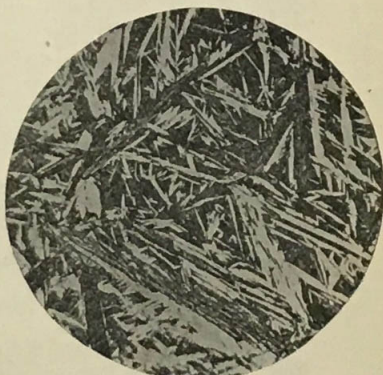


Fig. 40.

Steel cooled to  $-40^{\circ}$ ; 0.800 per cent C, 15 per cent Ni. Magnified 300 diameters. The photograph shows some acicular crystals colored white and black.



any transformation in cooling to  $-78$  degrees. In the case of steel having a martensite structure, however, this structure is apparent after cooling without etching.

## VI. CEMENTATION AND DECARBURIZATION OF NICKEL STEELS

It seemed to us interesting to confirm the results obtained in the experiments just described by means of some tests on cementation and decarburization. Of the many experiments conducted I shall only describe those of special interest.

These cementation experiments were made in the following conditions:

Temperature of the furnace: 1050 degrees.

Cement used: bone charcoal.

Length of the operation: 4 hours.

Diameter of the bars used: 20 millimeters.

Penetration of the carbon: about  $2\frac{1}{2}$  millimeters.

Slow cooling in the furnace itself.

The surface of the samples absorbed about 1 per cent of carbon. Much care must be exercised in conducting these experiments in order not to dull somewhat the edges of the samples. It is important to avoid this because the edge is precisely the most interesting portion. Mr. Le Chatelier suggested to weld together two samples so that only one surface needs to be polished and the edge of the weld can be readily observed.

1. *Cementation of steel containing 0.12 per cent of carbon and 2 per cent of nickel.*

The structure obtained is identical to that of ordinary carbon steel, and this is in accordance with results previously obtained; for it is well known that steel containing 2 per cent of nickel, even with 0.8 of 1 per cent of carbon, is constituted like ordinary steels.

2. *Cementation of steel containing 0.12 per cent of carbon and 7 per cent of nickel.*

In the centre, this steel is always formed of pearlite and of iron. Pearlite then increases and martensite begins to appear. Little by little acicular crystals are formed, which become more numerous as we approach the outside of the bar. This structure confirms the inference that martensite crystals seem to affect the shape of the acicular crystals in steel highly carburized. It is

also the result reached by Mr. Osmond in his experiments dealing with the quenching of carbon steel.

3. *Cementation of steel containing 0.12 per cent of carbon and 15 per cent of nickel.*

In this sample martensite is formed in the centre. Acicular crystals are next formed, increasing in number. A white portion is next observed which undoubtedly is made up *Gamma* iron. The surface of the steel contains about 1 per cent of carbon, and it is known that nickel steel containing 0.8 per cent of carbon and 15 per cent of nickel has a polyhedric structure. It is surprising that this layer should be so thin although the carbon content reaches 0.8 per cent to a considerable depth.

The explanation of these facts is now well understood. We know that upon reheating a sample of steel containing 0.8 per cent of carbon and 15 per cent of nickel this steel assumes a martensite structure. In the region containing 0.8 per cent of carbon, therefore, we shall have martensite because of the reheating which takes place at the temperature of the cementation operation, and the only portion in which a polyhedric structure can be formed will be that portion containing more carbon. As an additional evidence of the correctness of this conclusion it may be said that in a sample of cemented steel cooled to  $-78$  degrees no change of structure takes place, while if any steel with 0.80 per cent of carbon, 15 per cent of nickel remained which had not been transformed by the reheating, the thickness of the layer of acicular crystals should increase and the white portion should diminish.

4. *Cementation of steel containing 0.12 per cent of carbon and 20 per cent of nickel.*

The results are similar to those just described, but the polyhedric layer increases a little. This was to be expected, because the steel containing 1 per cent of carbon and 20 per cent of nickel is not transformed by reheating to 900 degrees for four hours.

5. *Cementation of steel containing 0.12 per cent of carbon and 30 per cent of nickel.*

The structure here becomes polyhedric all through the mass and indeed we have seen that reheating had no effect upon steel containing 0.8 per cent of carbon and 30 per cent of nickel. It is seen that the results of the cementation experiments agree with the results of all the other tests.

It may happen that by cementation, steel which is non-



magnetic becomes magnetic, and this is due not to the action of the cementation but to the reheating which changes the polyhedric structure into the martensite structure in the case of some steels. This important fact coincides perfectly with Mr. Dumas' results. If steels containing 27 per cent of nickel and 0.12 per cent of carbon or 25 per cent of nickel and 0.3 per cent of carbon, for instance, were subjected to cementation they would become magnetic. In short, nickel steels undergo through cementation the same transformations as through reheating.

All experiments on the decarburization were conducted at 800 degrees during eight hours. Steel containing 0.9 per cent of carbon was used and the cement was pure oxid of iron.

1. Decarburization of steel containing 5 per cent of nickel: the phenomenon is the same as in the case of ordinary carbon steel.

2. Decarburization of steel containing 10 per cent of nickel: in the centre the structure remains unaltered; it consists of acicular crystals nearer the edge and of martensite on the outside.

3. Decarburization of steel containing 15 per cent of nickel: In the centre the structure is not polyhedric and this is due to the effect of reheating. The acicular crystals become more and more numerous at first, then decrease and finally martensite is formed near the edges.

4. Decarburization of steel containing 30 per cent of nickel: This sample is not much changed by the reheating and the primitive structure is preserved. Near the edges a few acicular crystals are detected.

To sum up, these experiments confirm the results previously obtained. They show that the structure made up of acicular crystals is similar to the martensite structure, the first one occurring, in steels containing much carbon.

## VII. EXPERIMENTS ON THE REGENERATION OF NICKEL STEELS HAVING UNDERGONE THE TRANSFORMATION

It has been seen that some steels with a polyhedric structure undergo a transformation by quenching, annealing, cold-working and cooling below atmospheric temperature. Experiments were conducted to regenerate these steels, but in no case was this possible.

## CONCLUSIONS

The micrographic study of nickel steels which we have just described clearly shows that the constituents of these steels are of the following:

1. Ferrite, pearlite and, of course, troostite and sorbite.
2. Martensite.
3. Acicular crystals which appear after etching sometimes white, sometimes black, although the reason for this phenomenon is not known.
4. Polyhedric grains undoubtedly corresponding to Mr. Osmond's iron.

Are these acicular crystals identical with martensite? We believe it to be so because even in cast steel acicular crystals are present when the steel contains enough carbon. It appears to be, in the case of these highly carburized steels, the form assumed by martensite; in other words, it is hardenite. It is, on the other hand, almost certain that these acicular crystals represent, whatever the percentage of carbon in the steel, the form assumed by iron when subjected to any kind of treatment. In quenching, reheating, or cold-working a steel susceptible of undergoing the transformation, we have never obtained martensite but acicular crystals except in a single case. Finally, we shall call attention to the fact that the presence of these constituents confer on steel the same magnetic properties as those produced by martensite. The most interesting result of this study is certainly the perfect coincidence which has been shown to exist between the mechanical properties and the microstructure. This clearly follows from the experiments I have just related, and it may be said that the metallographic study of special steels will make it possible not only to see more clearly into the constitution of these metals, but also to infer more quickly and more simply their mechanical properties. They will, perhaps, lead to metallography being finally taken up in steel works as an important guide in their daily work.

Similar experiments with manganese yielded results of the same character. I hope to be able to publish them soon, as well as the metallography of chrome steel.

Finally, it remains for me to say that if I have been able to complete these experiments I owe it to the assistance given me



by the Dion Bouton Company,\* to the kindness with which Mr. Le Chatelier has guided me, and also to the help of the largest steel works of France. I am especially indebted to Mr. Poucel, Mr. Dumas, to the steel works of Imphy, of Unieux, and of Montluçon.

## THE INFLUENCE OF SULPHUR AND MANGANESE ON STEEL †

By J. O. ARNOLD and G. B. WATERHOUSE

IN the *Journal of the Iron and Steel Institute*, No. II, 1901, p. 234 *et seq.*, was published a voluminous and valuable paper by Mr. Axel Wahlberg, Director of the Testing Institution of the Royal Technical High School at Stockholm. This memoir dealt with the properties of the extensive series of heat-treated steels prepared by Mr. Brinell. As Mr. Wahlberg remarked, the paper lacked the important evidence certain to be obtained from a micrographic analysis of the bars experimented upon. Hence, per favor of Mr. R. A. Hadfield, Mr. Brinell kindly forwarded his steels to one of us to be examined in the micrographic laboratory of the Sheffield University College. The full examination of the series must necessarily occupy years, and the present paper is an instalment covering only a small portion of the ground, but that part is perhaps the most interesting, because it clears up mechanical phenomena due to the micro-chemical relations of carbon, manganese, and sulphur in steel.

### CHEMICAL

The most startling results obtained in Mr. Brinell's research were connected with a steel which had the following analysis:

Element	Brinell's Analysis	Author's Analysis.
Carbon . . . . .	0.460	0.460
Silicon . . . . .	0.369	0.370
Manganese . . . . .	1.060	1.07
Sulphur . . . . .	0.560	0.540
Phosphorus . . . . .	0.055	0.043

\* I must also express my appreciation to Mr. Le Grix who has taken the beautiful photomicrographs accompanying this paper.

† *Journal Iron and Steel Institute*, No. I, 1903.

Taking the mean of the foregoing analysis it will be evident that the manganese alloyed with the iron and carbon is only about 0.15 per cent, whilst the sulphide of manganese,  $MnS$ , will be 1.47 per cent by weight, or about 2.78 per cent by volume. The volume percentage is, of course, that measured by micrographic analysis.

### MECHANICAL

The above steel rolled perfectly and was almost the most ductile of the series under the impact test. In tension it gave the following figures:

Brinell's Mark	Author's Mark	Permanent Set in Tons per Square Inch	Maximum Stress in Tons per Square Inch	Elongation per Cent on 5.5 Inches	Reduction of Area per Cent	Thermal Remarks
4815	A	20.57	39.36	16.75	24.10	As rolled
"	B	24.96	40.60	18.10	21.90	Heated to 350° C.; cooled in air
"	C	28.45	39.70	17.60	24.10	Heated to 1200° C.; cooled in air
"	D	27.75	43.60	15.80	21.90	Heated to 750° C.; quenched in water at 20° C.
"	E	42.78	55.40	0.90	4.60	Heated to 1000° C.; quenched in water

The above results are fairly satisfactory, excepting the reduction of area per cent, which is less than half that obtained from a corresponding low sulphur steel.

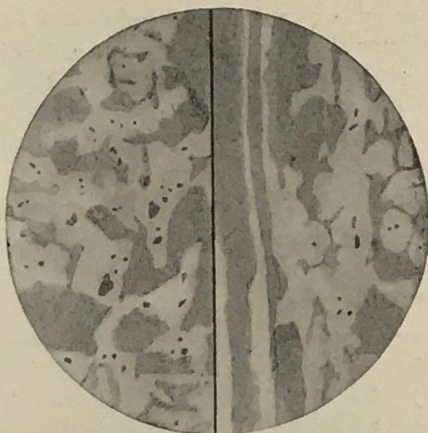
### MICROGRAPHIC

The sample marked B in the mechanical table is the rolled bar A heated to 350° C., and cooled in air. The micrographic structures of A and B are identical, being delineated in *Micrograph B*, and are as follows: The right half-section is longitudinal, and the left half-section transverse. It will be noted that both half-sections present three constituents:

1. Well-laminated pearlite.
2. White ferrite.
3. Dove-grey sulphide of manganese.

The remarkable feature of these sections, containing 0.46 per cent of carbon and over 1 per cent of manganese, is the fact that their





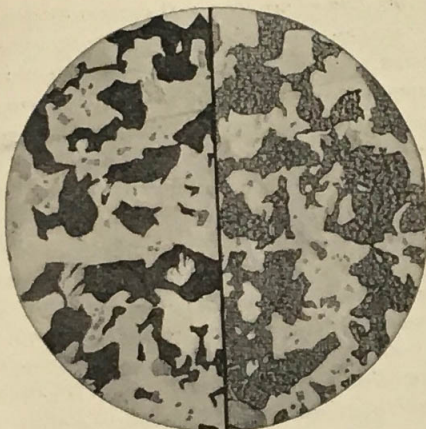
Micrograph B. Magnified 460 diameters.

Carbon	. 0.46 per cent	Permanent Set, tons per square inch	. 24.96
Silicon	. 0.37 "	Maximum Stress, " "	. 40.60
Manganese	1.07 "	Elongation per cent on 5.5 inches .	. 18.10
Sulphur	. 0.54 "	Reduction of Area per cent . .	. 21.90



Micrograph C. Magnified 460 diameters.

Carbon	. 0.46 per cent	Permanent Set, tons per square inch	. 28.45
Silicon	. 0.37 "	Maximum Stress, " "	. 39.70
Manganese	1.07 "	Elongation per cent on 5.5 inches .	. 17.60
Sulphur	. 0.54 "	Reduction of Area per cent . .	. 24.10



Micrograph D. Magnified 460 diameters.

Carbon	. 0.46 per cent	Permanent Set, tons per square inch	. 27.75
Silicon	. 0.37 "	Maximum Stress, " "	. 43.60
Manganese	1.07 "	Elongation per cent on 5.5 inches .	. 15.80
Sulphur	. 0.54 "	Reduction of Area per cent . . .	. 21.90



Micrograph E. Magnified 460 diameters.

Carbon	. 0.46 per cent	Permanent Set, tons per square inch	. 42.78
Silicon	. 0.37 "	Maximum Stress, " "	. 55.40
Manganese	1.07 "	Elongation per cent on 5.5 inches .	. 0.90
Sulphur	. 0.54 "	Reduction of Area per cent . . .	. 4.60



structures present that of an iron and carbon steel almost devoid of manganese; that is to say, the ferrite and pearlite are in almost equal proportions, and the latter is decisively laminated. In an ordinary manganese steel, as will be presently shown, the pearlite is not laminated, and is apparently (but only apparently) much greater in area than typical iron and carbon pearlite. The abnormal structure of this steel is, of course, due to the fact that the half per cent of sulphur present has appropriated practically the whole of the manganese to form  $MnS$ . This constituent Mr. Wahlberg anticipated would, on micrographic analysis, be discovered as a network. This view, however, is not confirmed by the microscope; in fact, had the sulphide of manganese formed sectional meshes, indicative of solid geometrical membranes, the material must necessarily have been both hot and cold short. It will be seen that the sulphide has been drawn out into long strings, presenting irregular transverse sections.

*Micrograph C* shows the steel heated at  $1200^{\circ}C$ ., and cooled in air. It exhibits precisely the same micrographic constituents as *Micrograph B*. The sulphide of manganese is unaltered, but the ferrite and pearlite have arranged themselves in a much smaller pattern.

*Micrograph D* exactly reproduces the structure of the steel quenched at  $750^{\circ}C$ . The left half-section shows the structure just as etched by Mr. Igevsy's reagent of alcohol, containing 5 per cent of picric acid, the best reagent yet proposed for etching steel. This left half-section shows apparently three constituents, namely, laminated pearlite, sulphide of manganese, and a dark-brown, amorphous-looking substance. The last named is Mr. Osmond's so-called constituent, troostite.

The right half-section shows the structure after gently rubbing with fine kid-skin. It now appears as a mixture of small brilliant particles of  $Fe_3C$  on a dark ground mass. This substance is not entitled to specific rank as a constituent or "ite," because it is an indefinite mixture of incipient pearlite and untransformed hardenite. The latter constituent deposits hardening carbon on etching, and so presents to a superficial examination the appearance of an amorphous constituent.

*Micrograph E* shows the steel quenched from  $1000^{\circ}C$ . Its structure presents dove-grey patches of sulphide of manganese, a few large and many small areas of structurally free ferrite on

a dark ground mass, very faintly showing a martensitic structure due to innumerable alternating plates of ferrite and hardenite.

### THE EFFECTS OF VARYING PROPORTIONS OF SULPHUR AND MANGANESE

It being evident that the relatively good mechanical properties of Mr. Brinell's high sulphur steel were due to the large proportion of manganese therein, the authors undertook further experiments to gauge the mechanical and micrographical effects of varying quantities of sulphur and manganese, preparing for this purpose three ingots similar to Brinell's steels.

The following table embodies the chemical compositions and forging capabilities of these ingots:

Ingot No.	C.	Si.	Mn.	S.	P.	Remarks
786	0.39	0.34	0.10	0.42	0.04	{ Ingot hopelessly cracked under the third blow of hammer. Ingot hopelessly cracked under the tenth blow of hammer. Ingot hammered and rolled perfectly to 18 mm. round.
787	0.35	0.32	0.45	0.47	0.04	
796	0.47	0.38	1.04	0.02	0.04	

*Table Showing Sulphide Percentages by Weight and by Volume*

Ingot No.	Per Cent FeS.		Per Cent MnS.		Remarks
	By Weight	By Volume	By Weight	By Volume	
Authors', 786	0.98	1.63	0.16	0.30	{ Would not forge " Forged well "
" 787	0.57	0.98	0.72	1.05	
" 796	None	None	0.054	0.102	
Brinell's, 4815	"	"	1.47	2.78	

The foregoing table shows in sharp relief the fatal effect of sulphide of iron on steel compared with the small effect produced by sulphide of manganese.



## MECHANICAL

It will be seen that the authors' ingot, No. 796, and Brinell's ingot, No. 4815, are identical in chemical composition except with reference to sulphur, 4815 containing sulphur 0.55, and 796 only 0.02 per cent of sulphur. The following table shows not only the great difference in mechanical properties in these two steels, but also the influence of very slow cooling on the properties of 796, which was heated for some time to 950° C., and allowed to cool for about twenty-four hours:

Steel No.	Treatment	Permanent Set. Tons per Square Inch	Maximum Stress. Tons per Square Inch	Elongation per Cent	Reduction of Area per Cent
4815	As rolled	20.6	39.4	16.8 *	24.1
796	"	40.8	52.2	25.0 †	49.9
796	Annealed	14.9	43.1	25.0 †	42.0

\* On 5.5 inches.

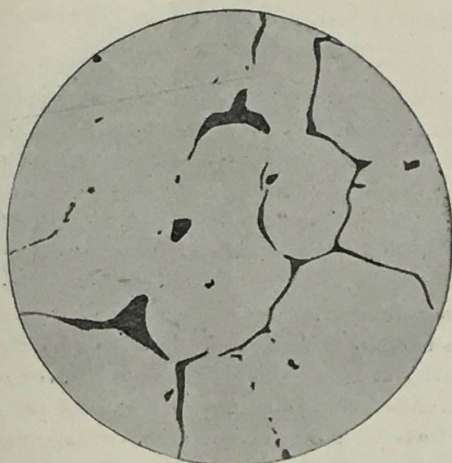
† On 2.0 inches.

## MICROGRAPHIC ANALYSIS

The micrographic analysis of 4815 has already been dealt with; it remains to describe the structures of ingots 786 and 787 and of the rolled bar obtained from 796 before and after annealing. A study of these structures will clearly explain the varying influences of both sulphur and manganese on steel.

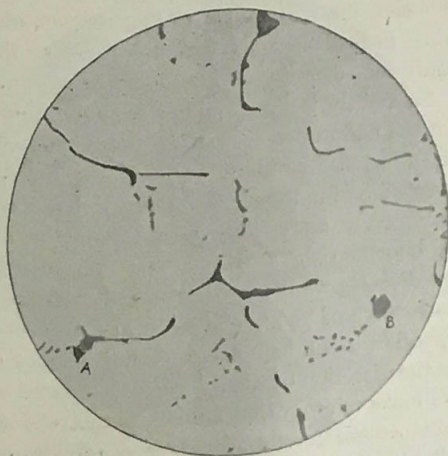
*Micrograph F* is 786 unetched. It exhibits the sulphide of iron as a pale-brown mesh-work, together with some irregular detached patches of the constituent. This micrograph is diagrammatic to the extent that where pieces of sulphide of iron had dropped out during the polishing (leaving dark cavities filled with rouge) they have been reproduced as though still *in situ*.

*Micrograph G*, also unetched, shows both dove-grey sulphide of manganese and pale-brown sulphide of iron; the latter is chiefly in the meshed form, whilst the sulphide of manganese is mainly in detached patches. In the areas marked A and B occur patches of sulphide of manganese and sulphide of iron closely juxtaposed, but quite distinct.



Micrograph F. (Unetched.) Magnified 460 diameters.

Carbon	. . .	0.39 per cent	Manganese	. . .	0.10 per cent
Silicon	. . .	0.34 "	Sulphur	. . .	0.42 "



Micrograph G. (Unetched.) Magnified 460 diameters.

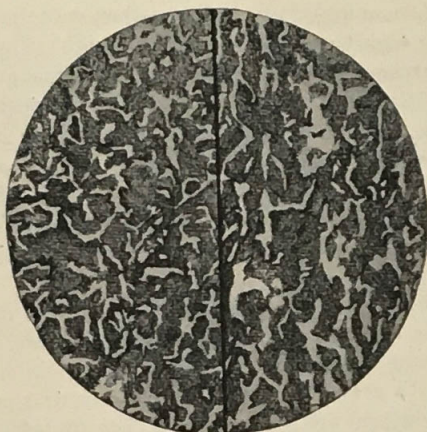
Carbon	. . .	0.35 per cent	Manganese	. . .	0.45 per cent
Silicon	. . .	0.32 "	Sulphur	. . .	0.47 "



CONSIDERATION OF THE CAUSES OF THE DIFFERENT EFFECTS  
OF THE TWO SULPHIDES

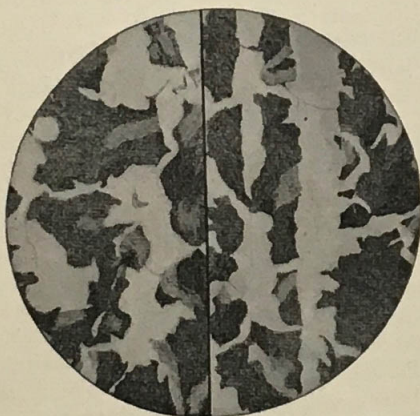
As has already been pointed out by one of us (*Journal of the Iron and Steel Institute*, No I, 1894, pp. 107 *et seq.*), the pale-brown sulphide of iron forms cell walls fusible at a bright-red heat, and hence renders the mass hopelessly red-short. Also the cell walls of sulphide, in solidifying at about  $950^{\circ}$ , contract from the cells of iron they envelop, and leave the mass cut into pieces by innumerable cracks. In the paper above quoted it was shown that iron containing about 1 per cent of sulphur had a tenacity of only  $2\frac{1}{2}$  tons per square inch, and owing to contraction cavities a specific gravity of only 7.69. On the other hand, the steel of Brinell and Wahlberg shows that steel with 0.55 per cent of sulphur in the form of dove-grey sulphide of manganese forges perfectly and gives good mechanical tests. In a paper on "Sulphide of Iron, its Properties and Condition in Iron," Professor Le Chatelier and Mr. Ziegler (*Bulletin de la Société d'Encouragement*, September, 1902) suggest that sulphide of manganese segregates at a high temperature. There is no reasonable doubt as to the correctness of this view. The falling out of this constituent at a white or yellow heat determines its segregation into globular masses upon which the main mass of the iron or steel contracts, and, therefore, instead of contraction fissures, there remains something equivalent to minute blow-holes filled with sulphide of manganese. So tightly is the sulphide of manganese held by the contraction of the mass upon it that it seldom falls out during the polishing, like the loose sulphide of iron. It is, however, quite evident that sulphide of manganese is fully plastic at a rolling heat, because the relatively huge, long streaks of this constituent in Brinell's steel show no signs of cracking or splitting.

*Micrograph H* shows the left half-section a transverse delineation of the structure of 796. The right half-section is the longitudinal view, and in it will be noted a slight tendency of the ferrite to elongate in the direction of the rolling. Compared with Brinell's 4815 this steel looks micrographically to contain about 0.7 per cent of carbon, because the ferrite areas are relatively much smaller, and the pearlite is all finely granular. These appearances are due to facts to which attention has been repeatedly called by one of us, namely, that manganese powerfully retards the segregation of



Micrograph H. Magnified 460 diameters.

Carbon	. 0.47 per cent	Permanent Set, tons per square inch	. 40.80
Silicon	. 0.38 "	Maximum Stress "	. 52.20
Manganese	1.04 "	Elongation per cent on 2 inches	. 25.00
Sulphur	. 0.02 "	Reduction of Area per cent	. 49.90



Micrograph I. Magnified 460 diameters.

Carbon	. 0.47 per cent	Permanent Set, tons per square inch	. 14.90
Silicon	. 0.38 "	Maximum Stress, "	. 43.10
Manganese	1.04 "	Elongation per cent on 2 inches	. 25.00
Sulphur	. 0.02 "	Reduction of Area per cent	. 42.00



ferrite and the lamination of pearlite — phenomena accounting for the relatively high tenacity of steel fairly high in manganese, in which the hardening carbon has, nevertheless, changed to cement carbon. To these causes, and not to varying positions of the change point, suggested by Mr. Wahlberg, are to be attributed those variations in hardness which he was inclined to consider as due to varying transformation temperatures. It has been exhaustively proved by one of us that the change point in pure iron and carbon steels is identical in position in steels varying in carbon from 0.1 to 1.8 per cent.

*Micrograph I* shows the remarkable change in micrographic structure from *Micrograph H* brought about by very slowly cooling another sample of steel 796. Such micrographic change amply accounts for the great difference in the mechanical properties of this steel before and after annealing. The left half-section was taken transversely, the right half-section longitudinally. It will be seen that the ferrite and pearlite are now in their normal proportions for a 0.46 per cent carbon steel, and that the pearlite areas are here and there distinctly laminated. The longitudinal section exhibits the remarkable phenomenon first described by one of us (in a lecture on Steel Rails delivered before the Sheffield Society of Engineers and Metallurgists, on March 25, 1901), namely, that the ferrite has crystallized in relatively huge laminae along stress lines set up by the operation of rolling, which lines have survived the operation of drastic annealing. This fact was confirmed by Mr. S. A. Houghton, in a lecture delivered by him before the Institution of Marine Engineers, in April, 1902.

#### SUMMARY

The results of the authors' investigations show:

1. That sulphide of iron is deadly in its effect upon steel, whilst sulphide of manganese is comparatively harmless.
2. That the above facts are due to the fusibility, the high contraction coefficient, and the tendency of sulphide of iron to form cell walls or enveloping membranes surrounding cells of ferrite; whilst sulphide of manganese is much less fusible, segregates whilst the iron is at a high temperature, and so collects into rough globules, and very seldom into meshes.

3. That manganese retards the segregation of iron and hardenite, and that what is called pearlite in a normally cooled manganese steel is really a mixture of granular pearlite and unsegregated ferrite.

4. That the complete segregation of the ferrite in a manganese steel can be brought about by very slow cooling, but that such annealing injures the mechanical properties of the steel by lowering the maximum stress and the reduction of area per cent registered by the unannealed steel.

NOTE. — The micrographs illustrating this paper have been reduced to half size during reproduction.

## ELECTRIC RESISTANCE OF STEEL \*

By H. Le CHATELIER

RESEARCHES on the electric resistance of steels have hitherto been principally carried on with the soft steels used for telegraph wires. They have proven that the difference in resistance between two samples depends almost exclusively on the relative proportion of manganese.

I have thought that it would be of value to study more systematically the influence of the different bodies which may be found in steels, especially that of carbon.

I have used for this purpose bars 1 square centimeter in section and 20 centimeters long. They were heated for several hours at about 600° C. Their chemical composition has been determined at the School of Mines by Messrs. Moutonet and Goutal. The numbers in the following tables express specific resistances (cubic microhm-centimeter) :

### *Influence of Carbon.*

Resistance	Carbon	Manganese	Silicon
10	0.06	0.13	0.05
12.5	0.20	0.15	0.08
14	0.49	0.24	0.05
16	0.84	0.24	0.13
18	1.21	0.21	0.11
18.4	1.40	0.14	0.09
19	1.61	0.13	0.08

\* *Comptes Rendus Academie des Sciences.*—Translated in *American Manufactures*, February 12, 1903.



The electrical resistance grows therefore very decidedly with the percentage of carbon; its increase is on an average 7 microhms for 1 per cent of carbon in weight or 1.5 microhm for 1 per cent of carbon atom in 100 atoms of iron and carbon.

Annealed steels consist of interlaced scales of ferrite (pure iron) and cementite (iron carbide  $\text{Fe}_3\text{C}$ ). By extrapolation of the preceding results, we find for the resistance of these two constituents considered alone: ferrite, 9.5; cementite, 4.5.

### *Influence of Silicon.*

Resistance	Composition	
	C.	Si.
12.5	0.2	0.1
38.5	0.2	2.6
15.8	0.8	0.1
26.5	0.8	0.7
33.5	0.8	1.3
17.8	1.0	0.1
25.5	1.0	0.6
32.0	1.0	1.1

These numbers show on an average an increase of 14 microhms for 1 per cent of silicon in weight, or 7 microhms for 1 atom of silicon in 100 atoms of the alloy; an increase of resistance much greater than for carbon. We are led, by analogy, with what occurs in a great number of alloys, to conclude, that in steels silicon is not isolated in the state of definite silicide  $\text{FeSi}_2$ , but is found in a homogeneous state, solid solution or isomorphous mixture, like the carbon used in tempering.

*Influence of Manganese.* — It is well known that manganese considerably increases the resistance of steels. This metal, isomorphous with iron, forms with it homogeneous mixtures in every proportion. But these mixtures can exist under two allotropic conditions which are unequally magnetic, both possessing different electric resistances, as I have already shown in regard to the manganese steel of Mr. Hadfield with 13 per cent of manganese.

Resistance	Composition		
	C.	Mn.	Si.
17.8	0.9	0.24	0.1
22	0.9	0.95	0.1
24.5	1.2	0.83	0.2
40	1.2	1.8	0.9
66 magnetic	1	13.	0.3
80 non-magnetic			

These numbers in general show an increase of resistance of about 5 microhms for 1 per cent in weight or in atoms of manganese. For magnetic manganese steel obtained by reheating non-magnetic steel for two hours at  $550^{\circ}$ , this increase of resistance is no more than 3.5 microhms.

*Influence of Nickel.* — Nickel, which forms an isomorphous mixture with iron, increases the electric resistance of the latter considerably, but these steels, as I have shown previously, present, like manganese steel, two varieties, unequally magnetic and at the same time unequally resistant. The increase of resistance is extremely variable with the proportion of nickel or with that of carbon, and with the allotropic state of the alloy. For quantities of nickel of less than 5 per cent, this increase has varied from 3 to 7 microhms.

*Influence of Chromium, Tungsten and Molybdenum.* — The influence of these bodies on the increase of resistance of steels is very slight. It cannot be measured exactly for the percentages less than 3 per cent, which are the only ones I have studied. The constant presence of silicon and manganese causes, with percentages generally less than 0.5 per cent, an equal and even greater increase of resistance than that resulting from the presence of 10 times larger quantities of the three metals in question. The slight influence of these metals on the electric resistance of steels leads me to think that they are isolated within the metal in the state of definite combination.

This conclusion agrees with the results obtained by Mr. Carnot in the chemical study of steels of the same nature.



**STEEL CASTINGS AND THEIR TREATMENT\***

By W. M. CARR

THE art of producing steel castings may be divided into three heads: 1. The making or refining of the metal; 2. the preparation of the molds and the casting therein of the metal; 3. the final treatment of the finished casting. In ordinary practice the operations under the first and second heads receive the best care and attention in the hands of the manipulators so that the casting shall be of a given composition and of a solidity and trueness to pattern which those concerned consider about all that can be expected. The final treatment of the finished casting, it is to be regretted, is often ignored, or when observed much misunderstood by many manufacturers.

By the third step is meant a certain form of heat treatment usually termed annealing. Even though the first and second steps be conducted skillfully, the good work accomplished can be destroyed by failure to anneal properly or by not annealing at all. Let a steel casting be sent out by any manufacturer without due observance of a suitable heat treatment, he yet has no assurance that in regard to its physical properties or its ability to meet the requirements expected of it in service, it is in the best condition to give satisfaction.

A large percentage of failures due to brittleness can be readily traced to a definite cause. Brittleness is always accompanied by a large coarsely crystalline structure when normal in composition. Such a structure is purely the result of a thermal condition at some stage of the manufacture. When a steel casting is desired for any purpose there always goes with it a thought of stability, of strength, endurance—a resistance to wear not known to the same degree in a gray iron casting. Yet how often do steel castings fall short of such an ideal condition. To be ideal the casting should be solid. The metal should be of sufficient toughness to withstand shock. In other words it should possess the maximum degree of strength combined with the maximum degree of ductility. It need not be considered a rule that as the strength increases the ductility will decrease, at least in dealing with commercial cast steel.

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\* *The Foundry*, September 1902.

In a great measure the composition and mode of manufacture will determine the ultimate strength, but the maximum ductility or toughness will depend upon the final heat treatment. An attempt to combine the ideal qualities and ignoring or imperfectly conducting the heat treatment will produce a metal which will be nothing other than a low-grade cast iron. Pot-metal would better express it, when it is possible easily to snap a hard, unannealed casting by a blow from a hand hammer. Failing to secure such qualities the maker has turned to softness resulting in a low composition chemically. The aim was to confer ductility, but sight was lost of the prime feature of a steel casting, i.e., strength. A casting so made may machine easily, but its wearing properties will be little better than those of its half-brother, wrought iron. A coupon cut from such a casting unannealed may show considerable elongation and contraction of area, but this is no argument against annealing any or all steel castings, though such evidence of ductility is often so used. It is true soft castings are frequently unannealed, but the writer contends that if softness or ductility is desirable, the maximum percentages of elongation and contraction of area, indices of ductility in any case, can only be gained by careful heat treatment. The fact of castings failing after annealing when used as an argument against such treatment will not hold in the light of advanced methods of heat treatment when aided by pyrometry and microscopy.

In many cases failures can be traced to improper treatment in annealing. Hence such a process requires some study, as does the art of melting or molding. It may be said that the three steps as previously outlined are one as important as the other, to combine the best results in a finished steel casting; consequently those who oppose annealing had better be sure of their ground in discussion of the question.

Let a case of failure in the steel casting be analyzed. It is assumed that the casting is returned broken after a comparatively short time in service. The fracture gives no evidence of blow-holes. It is a clean break; that is, has the characteristic metallic color in the absence of scale or oxidation (not rust). Then the failure is plainly not due to a hot or shrinkage crack. The next step, then, is to make a chemical examination of borings taken near the point of fracture. The subsequent analysis shows nothing in excess which might cause brittleness; that is, the metalloids are



within established limits. No light so far — an example of the limitation of a chemical analysis. Let the examination of a fracture be carried on a little more closely. The grain of the metal to the eye is large and glassy. The casting is not disturbed, hence the metal is brittle and must have been subjected to either a steady pull beyond its ultimate strength or has been given a heavy blow. Such evidence points to the lack of proper heat treatment, and a subsequent microscopical examination of the fracture as described will show how the metal was treated after casting; whether not annealed or improperly annealed. The remedy in such a case, or rather the prevention of a recurrence, necessarily entails a study of the distinct relations between the physical properties, structure and heat treatment.

The theory of a growth in the crystalline structure of a steel casting caused by continued vibration leading up to a rupture does not hold, since it is now known that the size and arrangement of the crystals is purely a function of temperature and rate of cooling between the melting point of steel and its critical range — a range several hundred degrees above atmospheric temperature.

As a rule steel castings are made from open-hearth metal, some preferring the acid steel to the basic. However, first-class castings can be made by the basic process as readily as by the acid. The point is this: It cannot be said that one rule of practice will apply to all. Some acid steel makers claim annealing is not necessary. The same argument is advanced by the users of basic steel. But to produce first-class castings of any variety made by any process, first establish a composition calculated to meet the requirements or purpose of the purchaser. Then as a protection to him, as a bulwark to the growth, reputation, future and establishment of the steel-casting industry, study the heat treatment in conjunction with the preliminary stages of manufacture. Then the finished casting made by such methods will be in the best possible physical condition when leaving the hands of the manufacturer. Otherwise the making of steel castings is but a leap in the dark.

In conclusion, a tabulation of physical tests of castings made in every-day operations is herewith embodied. Attention is called to the high tensile strength and the high percentages of the elongation and reduction of area in annealed specimens, an unusual feature in unforged steel. There is also given the Standard Specifications for steel castings as adopted by the American Section of the International Association for Testing Materials.

## STEEL FOR CASTING

C., 0.27, Man. 0.85, Si., 0.35, S., 0.020, Phos., 0.025.

*Physical Tests*

Tensile Strength	Elongation, %	Reduction of Area, %	
84,645	11.7	12.2	} Raw.
71,230	15.6	13.3	
88,126	19.5	23.1	
77,540	6.25	—	
92,465	24.2	38.3	} Annealed.
78,506	27.4	32.5	
69,595	32.0	42.2	
74,505	25.0	48.8	
71,830	11.0	14.6	} Improperly Annealed.
66,803	10.9	11.4	
86,340	18.8	22.0	
92,714	18.7	13.4	

As a knowledge of heat treatment grows there will be a corresponding increase in the percentages of elongation and reduction of area over those given in the present standard specifications.

## STANDARD SPECIFICATIONS FOR STEEL CASTINGS

Adopted by the American Section of the International Association for Testing Materials.

	Tensile Strength	Elongation, %	Reduction of Area, %
Hard Castings . . . . .	85,000	15	20
Medium . . . . .	70,000	18	25
Soft . . . . .	60,000	22	30



## THE PREPARATION OF SAMPLES FOR MICROSCOPIC ANALYSIS AS FOLLOWED BY THE WESTINGHOUSE ELECTRIC AND MANUFACTURING COMPANY \*

By W. J. BECK

THE principal work done in the microscopic study of metal has been with low carbon sheet steel (used for electrical purposes), although some attention has been given to cast iron, cast steel, cold rolled steel, and alloys of babbitt and brasses.

In preparing a sample for a photograph, three distinct operations are followed.

First. — Cutting and shaping of the sample.

Second. — The polishing and etching of the surface to be photographed.

Third. — Microscopical examination and photographing.

*Cutting and Shaping of the Sample.* — In the case of sheet steel analysis, the sheet of a known heat or lot is selected, and strips of approximately  $\frac{1}{4}'' \times 1''$  are cut from the various parts of the sheet, and each is recorded, and at once stamped with a serial number. After having selected several samples, they are taken, one by one, and mounted on a small holder. This holder consists of a block of hardened steel  $\frac{1}{4}'' \times \frac{1}{4}'' \times \frac{1}{2}''$  with perfectly squared sides. Over this is a small frame with internal dimensions  $\frac{1}{8}'' \times \frac{1}{4}'' \times \frac{5}{16}''$ , and provided with small thumbscrews for fastening the samples when in position. In forming the sample on the holder, it is first clamped in the vise between the holder, and a second piece of hard steel in order to obtain a smooth surface for polishing. It is then bent down on the sides of the holder forming a U-shaped piece or sample. It is then removed from the vise, and a clamp put on, holding the sample in position with the thumbscrews. This gives a perfectly flat surface on top and bottom of the holder, and the sample need not be removed for examination under the microscope during the process of polishing and photographing.

*Polishing and Etching of the Samples.* — The apparatus used for polishing samples of metal is similar to the apparatus

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\* *Proceedings of Engineers' Society of Western Pennsylvania*, December, 1902.

described by Mr. Albert Sauveur in *The Metallographist* of October, 1901. [This polishing machine consists of four polishing wheels mounted on a horizontal shaft and revolving in a vertical plane. The first wheel is a solid fine-grain emery, approximately 8" in diameter. The second is a metal disc covered with coarse canvass coated with an emery paste. The third and fourth are made up of a wood block, the third being covered with a good quality of billiard cloth, and coated with a white paste, consisting principally of tripoli and soap. The fourth is covered with a heavy broadcloth, for which is used a paste made of jeweler's rouge. An average of twenty samples have already been prepared without renewing the cloth on the wheel, which can be done in a very short time.]

Having mounted the sample on the holder, it is not necessary to do any filing on the surface to be polished, there being but a thin coat of oxide on the surface which can be easily removed with the emery wheel, thus giving a more true surface than that obtained by means of a file or by hand on emery cloth.

When applying the sample to the emery wheel, care is taken not to hold too long in one position, but gradually to keep changing to avoid cutting deep furrows in the surface. One or two slight touches of the sample on the emery wheel are generally sufficient to remove the scale or oxide. It is then passed to the second wheel, and the heavy scratches removed with the emery paste. The sample is then washed thoroughly and passed to the third wheel. In most cases, and for ordinary examinations of the sample, a sufficiently good surface is obtained from this wheel. If, however, photographs are to be taken, the sample is again thoroughly washed, then applied to the fourth wheel on which a paste made of jeweler's rouge is used with an abundant supply of water, in order that the rouge does not become hard and scratch the surface.

*The Etching of the Sample.*—The method followed in etching a soft steel sample is one in which it is possible to carry on the etching of several samples at one time. Various methods have been tried, but the results as thus far obtained have been with the use of a solution of diluted nitric acid. The strength of this solution is approximately one part nitric acid and twenty parts water. The time required to give a properly etched surface varies from  $\frac{1}{2}$  to 3 minutes, depending upon the hardness of the steel.



*Microscopic Examination and Photographing of the Samples.*

— The apparatus used for the examination, and photographing of the samples is a Bausch & Lomb microscope with camera attachment, and fitted with apochromatic objectives with compensated eye pieces and a mechanical stage.

The light used is from a D. C. arc lamp, automatically fed, and fitted with condensers and cooling cells, giving parallel rays which are concentrated by bull's-eye through a ray filter, and then thrown on the illuminator of the microscope.

In the study of the structure of the metal, the image is generally thrown on a screen, which makes it much easier to select the part desired for the photograph, and less tiresome to the eyes. Having selected the section to be photographed, the plate is exposed from one to three seconds, depending upon the surface of the sample, and quantity of light admitted. By the use of the ray filter, much more satisfactory results have been obtained, and it is thought that in this way less expensive objectives may be used with very good results. All photographs are taken at about 80 diameters for convenience in comparison of the structure.

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## TESTS ON FINISHING AND ANNEALING HEATS \*

By F. C. LAU

THE experiments herein set forth are not laboratory experiments, but experiments conducted along the regular line of shop practice, and the deductions have been drawn from the work performed by many tools, covering a period of nearly two years.

The question which prompted the investigation was, why the life of tools should vary to a considerable extent, all doing the same class of work, as in the case of a set of punches. Feeling satisfied after examining many of the short-lived punches that the cause was not due to improper hardening, I turned my attention to the annealed structure. Bars were selected of different structures from a fine to a coarse grain.

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\* *Sparks from the Anvil*, October, 1902.

After the punches had been finished, except hardening, they were polished and etched, examined under the microscope, and graded according to their structure. Each punch was numbered for the purpose of keeping a complete record. Frequently I found it necessary to divide a single six or seven-foot bar into three or four structural grades. It appears that the differences in structure in the same bar are due to the working heats, rather than to the annealing.

The fatigue test under heavy duty in punching is no doubt the most reliable test to which the hardened structure can be

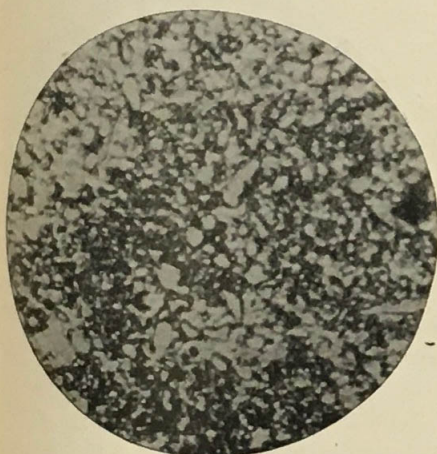


Fig. 1.

C 1.15 per cent. Properly annealed. Polished on a plain surface. Magnified 1,000 diameters.

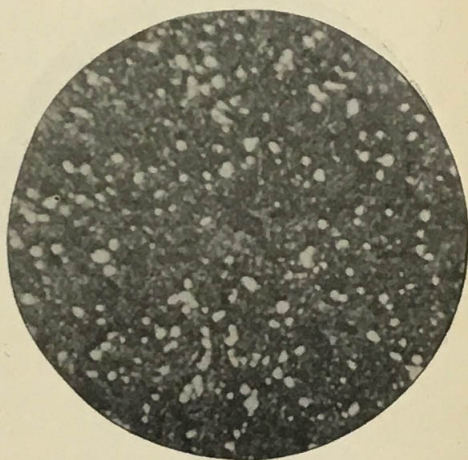


Fig. 2.

C 1.50 per cent. Hardened from AC<sub>1</sub>. Polished on a plain surface. Magnified 1,000 diameters.

subjected; the results remain constant as long as the structural grade remains the same.

Three thousand one hundred and sixty-two punches have been subjected to the test in the regular line of work. Punches of three or four structural grades were arranged alternately in each set, in order to get a fair test, and in every test it was found that the annealed structure, as shown in photomicrograph Fig. 1, and the hardened structure, as exhibited in Fig. 2, gave the best results.

Four sets of like structure were tested separately upon the same class of work with uniform results, hence I have called the



work done by such structure 100, and used that as a base for calculating the work of the other structures. It might be stated here that all structures were hardened from  $Ac_1$  and the temper was drawn to the lowest point where the resistance to impact remained perfect. If a higher heat is used in hardening, or the punch left too hard, the percentage of work done will fall below that of the  $Ac_1$  hardening, etc.

Fig. 3 reveals the structure of a steel annealed from  $900^\circ$  C. Upon a closer inspection we notice that parts of three crystals are



Fig. 3.

C 1.14 per cent. Annealed from 900 degrees C.  
Magnified 1,000 diameters.

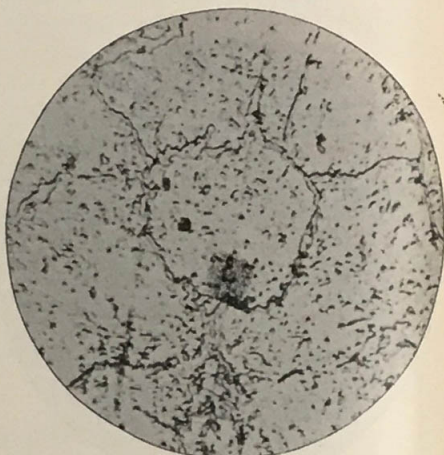


Fig. 4.

C 1.39 per cent. Hardened from  $AC_1$ . Combined  
polished and etched with sodium chloride; this  
method only develops the cementite.  
Magnified 1,500 diameters.

shown, with a wall of cementite between them. The work done by this structure fell 16 per cent short of that of Fig. 1. Tests were also made of annealings from  $750^\circ$ ,  $800^\circ$  and  $850^\circ$  C. The results revealed the fact that for every  $50^\circ$  added above  $700^\circ$  C. there is a loss of 4 per cent in work performed.

Fig. 4 shows the structure of a bar where the finishing heat was too high for the amount of work. The work done upon the bar affected the structure to a depth of about  $2\frac{1}{2}$  mm., leaving the interior a large crystalline structure with the cementite enveloping each crystal, "a source of weakness." Its loss was 3

per cent from the standard, and had it not been for the perimel structure which was equal to that of Fig. 2, its loss would no doubt have fallen to at least 15 per cent; thus we see how important it is to have a uniform structure throughout.

There are three points which have their influence upon the life of a tool:

- A. The finishing temperature.
- B. The annealing.
- C. The hardening.

The amount of work to be done upon a bar should govern the finishing temperature.

It is a well-known fact that at a high heat, carbon, phosphorus, and sulphur become mobile and will segregate in large patches which become a source of weakness, and for this reason the work should be heavy enough to check and break up the segregation, and also for the purpose of retaining a uniform structure throughout the mass. It must also be remembered that the higher the heat the larger the crystals, unless they are checked in their growth by the work upon the bar.

Not enough attention is paid to the finishing temperature. It is taken for granted that annealing will correct the error made in the finishing heat. All experiments fail to support the theory. Again, as in the case of milling cutters, lathe and planer tools, here the results fall 20 per cent below the standard, or in other words, a tool made from a bar whose structure is represented by Fig. 1 and Fig. 2 will do 20 per cent more work than a tool with a structure as shown in Figs. 3 and 4.

In the case of milling cutters all experiments point to the fact that such tools should be made from blanks made for that purpose, and not cut from large bars. The blanks present a uniform structure while the bars do not, unless the blank from the bar has been properly worked before being made up.

*Annealing.* — By the term annealing it should not be understood to mean that the steel must be subjected to a white heat or to a soaking heat of two or three hours and then slowly cooled. Many valuable tools have their values cut as high as 20 per cent in this operation, and when the tool fails to hold a cutting edge, and “flakes out here and there,” notwithstanding the fact that it has been properly hardened, it is finally returned to the steel maker with a note that the steel is “no good.” Can we expect



good results from a tool whose structure is equal to that of Fig. 4 and 5?

Fig. 5 illustrates the structure of a tool which received a soaking, annealing heat at about  $800^{\circ}\text{C}$ . Its work fell 11 per cent short of the standard. Here we notice the segregation of cementite due to the long heating. Such a structure will not hold a fine cutting edge, but will flake out.

The annealing heat should never exceed the  $A_{c1}$  point, or in other words, the proper "hardening heat."

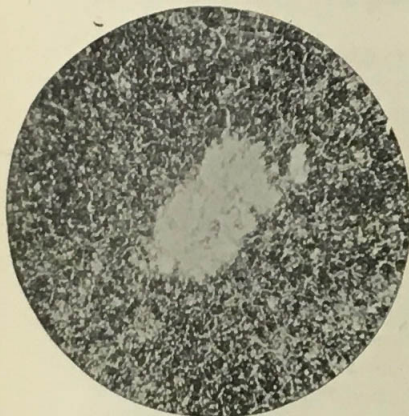


Fig. 5.

C 1.39 per cent. Annealed. Soaking heat and then hardened from  $A_{c1}$ . Magnified 150 diameters.

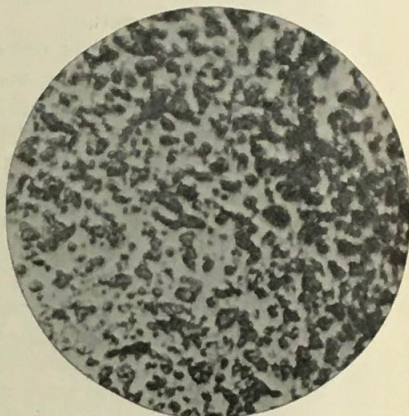


Fig. 6.

C 1.15 per cent. Properly annealed. Combined polished in relief and etched with sodium nitrate. Magnified 1,000 diameters.

A glance at Fig. 3 shows a laminated structure with the pearlite and cementite well defined. If the heat had been prolonged, the striations of cementite would have been much larger and hence a greater detriment to the steel.

Figs. 1 and 6 represent the annealed structure which gives the greatest value to a tool. In these two structures we find that the cementite has been changed *in situ*, thus leaving the structure an ill defined granular pearlite structure, and when such a structure is properly hardened, as shown in Fig. 2, we have the ideal condition of hardened steel. I have purposely selected a higher carbon steel to illustrate the properly hardened structure, and for the

purpose of showing that the cementite will remain in a finely divided state as long as we do not resort to drastic heat treatments.

The experiments have demonstrated the fact that in order to get the best possible results out of the steel the cementite must be diffused throughout the mass in as finely divided a state as possible, and this can only be accomplished by the use of a proper finishing and annealing heat.

The experiments have been made with the greatest care, and I am satisfied that the deductions are correct for pure carbon steel.

Analyses of twelve of the pieces gave the following average results, the carbons being as stated under the illustrations: Silicon 0.106, phosphorus 0.011, sulphur 0.009, manganese 0.20.

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## METALLIC CONDUCTION AND THE CONSTITUTION OF ALLOYS\*

By JOHN ALEXANDER MATHEWS

IN a paper recently presented by the author, before the Iron and Steel Institute,† this subject was touched upon to a slight extent, and it was stated therein that "the evidence thus far, though insufficient, leads me to hope that a law will be found connecting atomic concentrations and the specific conductivities of added metals with the resulting change in the conductivity of the principal metal. Before this can be possible, more must be learned about the intermetallic chemical relations of alloys." This opinion was based upon the recent work of Barrett, Brown and Hadfield,‡ Le Chatelier§ and the classic researches of Matthiessen, together with some recent measurements by myself. For the most part steel alloys have been studied. It was also pointed out that a study of quenched, rather than annealed materials, might be profitable, for if alloys are to be considered as solutions, it is probable that some atomic relation affecting their resistivity exists. Now

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\* *Electrical World and Engineer*, October 4, 1902.

† "A Comparative Study of Some Low Carbon Steel Alloys," *Journal Iron and Steel Institute*, No. I, 1902.

‡ *Sci. Trans. Royal Dublin Soc.*, vii, p. 67.

§ *Bull. Soc. d'Encouragement*, III, p. 743.



quenching tends to keep all the constituents of an alloy in a homogeneous condition, resembling that which existed while the alloy was molten, while annealing, particularly of steels, promotes segregation of the constituents in a way analogous to precipitation in ordinary solutions. The constitution of alloys has been very fully discussed in two recent papers by the author,\* and the perusal of either of them by readers not familiar with recent work upon this subject may help to make clear what follows.

Nearly simultaneously with the presentation of my paper to the Institute, there appeared in England a later paper by Prof. Barrett,† the distinguished physicist, whose name is associated with the discovery of the phenomenon of recalescence in steel, and one in Germany, by Mr. Carl Benedicks,‡ both of which tend to confirm in part the opinion quoted at the beginning of this article. At the same time, however, they lead to the conclusion that it is not a new law, but the extension of an old law to a field in which it was not known or supposed to apply — the law of Mendelejeff and Newlands that “the properties of the elements are a periodic function of their atomic weights.”

It will be remembered that in studying the effect of various elements on gold, Roberts-Austen§ found that the mechanical properties of that metal were, in general, changed in a manner proportional to the atomic volumes of the added element. The larger the atomic volume, the more deleterious was its effect upon gold. This law only applies to small percentages of added impurities, i.e., to those which may be supposed to yield a single solid solution, and its general applicability is further interfered with by the individual chemical behavior of the added metal toward the principal metal. The formation of a definite intermetallic compound by the two metals would naturally produce a different effect than a simple solution of one metal in the other, but such a condition in no way invalidates the law, for it was only supposed by its discoverer, Sir William Roberts-Austen, to apply to dilute, solid solutions. Osmond's work seems to show that the influence of elements on iron is in accordance with the periodic law. The

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\* “Alloys as Solutions,” *The Mineral Industry*, vol. X, or “Upon the Constitution of Binary Alloys.” *J. Frank. Inst.*, Jan.-March, 1902.

† *Proc. Royal Soc.*, LXIX, p. 480.

‡ *Zeitschrift für physikalische Chemie*, 1902, vol. 40, p. 545.

§ *Phil. Trans. of the Royal Soc.*, CLXXIX, p. 339.

close relation between the periodic law and Roberts-Austen's law of atomic volumes is seen when we recall that atomic volume = atomic weight  $\div$  specific gravity.

In Professor Barrett's recent paper, he points out a relation existing between the increase of electrical resistivity in iron alloys and the specific heat of the added element. But the specific heat multiplied by the atomic weight, in most cases, gives a constant; hence, the increased resistivity is as closely related to one as to the other. The alloys studied by Prof. Barrett were the very excellent series prepared by Hadfield, and included a large number of alloys of many elements with iron. In their earlier paper on the conductivity of these alloys, Messrs. Barrett, Brown and Hadfield state that "an approximate estimate of the quantity of carbon in any specimen of carbon-steel might be quickly obtained by a determination of the electrical conductivity of the particular sample, provided that the other constituents, especially silicon and manganese, were practically uniform throughout the specimens. On the other hand, from the electrical conductivity of samples of steel, in which the percentage of carbon only is known, we can infer the purity or otherwise of the samples." This very interesting observation might often be applied advantageously in testing materials.

In discussing the relation between conductivity and specific heat, Professor Barrett says: "From the conductivities of the specimens their specific resistances were calculated; these were plotted against the percentages of the added element. A series of fairly smooth curves were thus obtained for each alloy." There is obviously no connection between the curves thus obtained and the conductivities of the added metal — for example, aluminum produced the greatest effect and tungsten the least; and aluminum, which is a better conductor than nickel, produces a greater increase in resistance than nickel. Neither is there any connection — as Professor Barrett observes — between physical hardness and the conductivity; the hard manganese and tungsten steels are better than the soft silicon and aluminum steels for equal percentages of impurity. The following table, from his paper, shows the approximate increase in resistance (in microhms per cubic c.c. at 18° C.) of annealed iron by adding to it the metals named in the first column of the table:



TABLE I.—(BARRETT)

Alloys of Iron with:	Per cent of Added Metal		
	2.	3.	5.
Tungsten . . . . .	4.0	5.0	6.0
Nickel . . . . .	7.0	9.0	13.0
Chromium . . . . .	10.0	11.5	14.0
Manganese . . . . .	16.0	18.0	24.5
Silicon . . . . .	26.0	34.0	40.0
Aluminum . . . . .	28.0	36.0	54.0

From this table it will be noticed that the increase of resistivity decreases with each addition. In a second table, Professor Barrett shows the approximate increase of resistance for one per cent of added metal, and compares this with the specific heat of the added metal. I have modified this table by the omission of carbon and by the introduction of more recent values for the specific heats of chromium and aluminum, the new values in both cases agreeing more closely with those required by the law of Dulong and Pettit.

TABLE II.—(BARRETT)

Alloy	Inc. in Res. 1%.	Spec. Heat.	Atomic wt.
Tungsten . . . . .	2.0	0.035	184.0
Cobalt . . . . .	3.0	0.107	59.0
Nickel . . . . .	3.5	0.109	59.0
Chromium . . . . .	5.0	0.121	52.0
Manganese . . . . .	8.0	0.122	56.0
Silicon . . . . .	13.0	0.183	28.0
Aluminum . . . . .	14.0	0.227	27.0

It must be recalled that in dealing with percentages as high as those of the first table, we are not sure that the added metal remains in solution, especially in the annealed samples.

Some of Professor Barrett's curves are shown herewith. In the diagram I (Fig. 1) the added elements are expressed in ordinary percentages, as given by him originally. The second diagram shows the result of replotting them in atomic percentages, the calculations being made upon the basis of the number of atoms of added metal in 100 atoms of iron. The dotted portions of diagram (Fig. 2) have no connection with the subject immediately at hand.

In studying the relation of electrical resistance to the constitution of the conducting alloy, we must always bear in mind what a complex substance a steel alloy is: that it always contains carbon, manganese, silicon, sulphur and phosphorus; that the iron itself may exist in two or three allotropic conditions; that the limits of solubility of various elements in iron are in most cases unknown,

and that the intermetallic compounds of iron with another metal, or of two metals with carbon forming a double carbide, have been very imperfectly worked out.

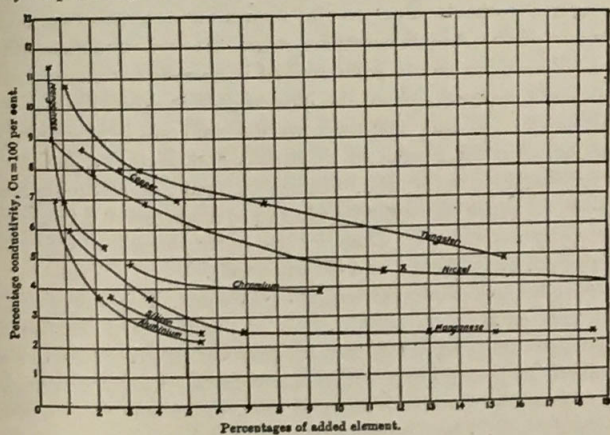


Fig. 1.

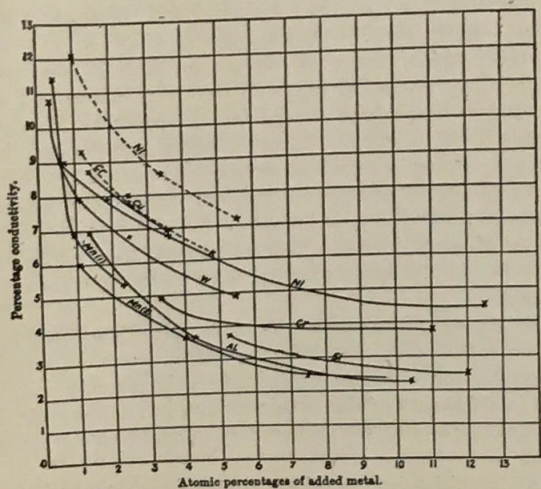


Fig. 2.



Yet, notwithstanding these complications, a broad view of the existing evidence leads to the idea that the atomic law is in some way connected with the problem. Professor Osmond, in discussing the author's paper to the Iron and Steel Institute, takes this view of the subject when he says: "It was remarkable that Mr. C. Benedicks should have arrived independently and simultaneously at the conception of the idea of atomic equivalents of dissolved bodies. It was true there were some considerable discrepancies between the results of Benedicks and Mathews, but there was nothing very surprising in these discrepancies, any more than in those on which the author (Mathews) commented as existing between results of M. Le Chatelier and other physicists in the case of tungsten, molybdenum and chrome-steels.

"If the law of atomic weights was exact, it was applicable, as its very enunciation implied, only to the dissolved fraction of the alloyed substances, but this fraction varied according to the treatment, and was unknown in many cases, notably chromium and tungsten, and probably molybdenum, too, would form double carbides, which, when liquated, would have but a very weak influence. Before concluding, it would be necessary to know in each particular case what was dissolved and what was not, and also what was the state of the substance in solution. These were questions to which one could but seldom find an answer, and much investigation, as Dr. Mathews rightly said, was still necessary." It was in the hope of stimulating further work upon this interesting problem that we venture to bring it to the attention of electricians, hoping — as we have elsewhere stated in regard to these views — to "provoke discussion, and, more especially, thought and research in this important field. Their confirmation would be of great interest; their refutation upon experimental evidence would doubtless produce some other and correct explanation."

At the time that remark was made, Mr. Benedicks, in the University of Upsala, was bringing forth the most conclusive evidence that for small concentrations the increase in resistivity of steel is a function of the atomic weight, i.e., equi-atomic solutions of metallic elements in iron produce equal increase in electrical resistance.

Mr. Benedicks determined the electrical resistance of a number of samples of steel which had been carefully analyzed. They

contained varying quantities of carbon, silicon, manganese, sulphur and phosphorus, the last two elements being low and fairly uniform. The steels were tested in both the hardened and annealed state. From his determinations, the author found that one atomic per cent of various elements dissolved in iron produces an increase in resistance which is equal to 5.9 microhms per  $\text{c.m}^3$ . The author also calculated that the resistance of absolutely pure iron would be 7.6 microhms per  $\text{c.m}^3$ , but this value is lower than has ever been obtained experimentally, for perfectly pure iron has not been investigated. By means of the following formula, it was found possible to calculate the resistance of steel with considerable accuracy:

$S = 7.6 + 26.8 \Sigma C$ , in which  $S$  = resistance in microhms per  $\text{cm}^3$ , and  $\Sigma C$  = % foreign substances calculated as equivalent quantities of carbon.

But in order that this formula be applicable, it was necessary to ascertain the effect of carbon itself upon the conductivity. This Mr. Benedicks has done very skillfully. It is now generally understood that carbon combines with iron to form a carbide,  $\text{Fe}_3\text{C}$ . This exists in annealed steels in a condition easily recognizable under the microscope, and is known to metallographists as "cementite." When steel is heated to temperatures above  $700^\circ \text{C}$ . and cooled suddenly, no cementite appears, but under the microscope, at high magnification, a structure known as "martensite" is seen. The carbon in this condition may be combined or simply dissolved without combination. Professor Arnold thinks that a sub-carbide,  $\text{Fe}_{24}\text{C}$ , exists in hardened steel.

However, when hardened steel is reheated and slowly cooled, cementite appears, and the other micrographic constituent is known as "ferrite." This has usually been considered to be *pure* iron, and the combined ferrite and cementite, when existing in alternate bands or layers, constitute the "pearly constituent" of Dr. Sorby, now called "pearlite." Mr. Benedicks shows that ferrite is not carbon-free iron, but that annealed steels, containing from 0.40 to 1.70 per cent carbon, consist of cementite and iron containing about 0.27 per cent of dissolved or hardening carbon. This constituent may be identical with the "sorbite" of Osmond. The segregated carbon in the form of cementite exerts little influence upon the conductivity. This statement does not seem to accord with that of Le Chatelier, who gives the resistance of ferrite as 9.5, and cementite ( $\text{Fe}_3\text{C}$ ) as 45.



When a low carbon steel — less than 0.40 per cent carbon — is annealed, cementite separates, but the accompanying ferrite contains less carbon than 0.27 per cent. For example, Mr. Benedicks states that the ferrite of a 0.20 carbon steel would contain 0.06 — 0.07 per cent of dissolved carbon. Benedicks' work confirms the chemical researches of Osmond and Werth, Carnot and Goutal, Brustlein, Arnold, and Stansfield in regard to the existence of 0.27 per cent of hardening carbon in solid solution in annealed high-carbon steels. All this work strengthens the author in the opinion he has held and expressed repeatedly, that in the separating or crystallizing out of the constituents of any alloy, no pure metal ever separates, but metal containing more or less of the other constituents of the alloy in solid solution. It seems, too, that in Benedicks' confirmation of the atomic equivalence of the elements in their effect upon conductivity, we have presented some new evidence in favor of the opinion expressed by Professor Ramsey, in 1889, "that it would appear legitimate to infer that in solution, as a rule, the atom of a metal is identical with its molecule."

As regards the exact nature of the phenomenon of conductivity in metals and alloys, very little is known. The beautiful theory of electrolytic conductivity in ordinary liquid solutions seems to have nothing in common with metallic conduction. In a way, these two kinds of conductivity seem to be opposed to each other, for while the conductivity of a pure liquid is increased by the presence of dissolved matter in it, a pure metal becomes a much poorer conductor if another element is added to it. Dewar and Fleming's researches showed that the resistance of pure metals decreased with falling temperatures, and there was evidence that at the absolute zero the resistance would be *nil*. In the case of alloys, no such evidence was afforded; they seem to follow some other law. What is true in regard to electrical conductivity probably holds for thermal conductivity as well. Kohlrausch showed that for high carbon steel, low carbon steel and wrought iron the ratio between these two kinds of conductivity is close.

The electrical resistance of steel at high temperatures furnishes one of the evidences of the allotropy of iron. Le Chatelier found two reversible transformations in the conductivity of a certain sample of malleable iron at 730° C. and 855° C. These temperatures correspond closely to the "breaks" in the cooling curve of mild steel, designated by Osmond as  $Ar_2$  and  $Ar_3$ , i.e.,

in slowly cooling such a sample of steel there occur two evolutions of heat at certain temperatures. These evolutions of heat occur in the purest carbonless iron. In iron containing carbon, their point of occurrence is affected by the presence of that element, but since its presence is not the cause of their occurrence, they have been thought to indicate molecular or allotropic changes in the iron itself. As has been shown for iron, the first additions of any other element produce greater increase in resistance than any subsequent additions. It is these first small additions that may be supposed to remain in solid solution, and to act most deleteriously upon the conducting power of the iron. Matthiessen showed that the conductivity curves for most pairs of metals — excluding the metals lead, tin, zinc and cadmium — is of the shape of the letter U. Alloys whose compositions lie along the part of the curve where it is nearly vertical, are those in which a single solid solution is most likely to occur. Intermediate compositions usually consist of at least two constituents crystallizing together in some sort of a conglomerate of solid solution, intermetallic compound, eutectic, etc. However they crystallize, each constituent is more or less saturated with the other at the temperature of solidification. All such alloys conduct poorly, but it is not known that the size of the grain or the number of boundary faces between contiguous grains influences the conductivity directly. The presence of such constituents, microscopically discernible, has suggested to Lord Kelvin the idea that the increased resistance may be due to thermo-electric effects, caused by the passing of the current. He says, "the temperature thus arising increases until the conduction of heat through the laminæ balances the Peltier effect at the junctions, and it gives rise to a thermo-electric force opposing the passage of the current." Now, if such effects take place at points of contact between laminæ of relatively large size and of different compositions, why might not the same phenomenon occur between much smaller laminæ, and why might they not occur between the atoms themselves — in other words, why will not the explanation hold good for solid solutions? This would account for a greater effect from a dissolved than from a segregated substance.

In the case of non-ferrous alloys, there is some evidence in favor of the existence of an atomic relation governing conductivity. These alloys would be the easiest to study by way of confirmation of the evidence afforded by steel, because they can be obtained



in a greater degree of purity. From existing data, however, something in favor of the atomic theory as applied to metallic conduction has been gathered. This we will summarize briefly in conclusion. The only two metals which we know to be perfectly soluble in each other, and which never separate into two microscopic constituents, are silver and gold. The highest degree of mutual solubility of which we can conceive would be afforded by an alloy of the composition Au-Ag, i.e., an equi-atomic alloy. It is precisely this one which has the highest resistance. Alloys whose compositions are represented by the atomic values  $Au_4Ag$  and  $Ag_4Au$  conduct about equally well. In the investigations of the Reichsanstalt upon the conductivity of nickel-copper alloys, it was found that the alloy consisting of almost equal atoms of nickel and copper had the highest resistance. In the case of copper-silver alloys this does not hold good, but, nevertheless, the solution theory in regard to resistance is confirmed. Matthiessen found the highest resistance of silver-copper alloys to be afforded by Levöl's alloy, which contains about 28 per cent of copper.

This is the one alloy of copper and silver which on slow-cooling in mass does not segregate, but is of uniform composition throughout. It is, in fact, the eutectic alloy, and a microscopic examination of it reveals the fact that it is composed of two constituents. One of these we believe to be a saturated solid solution of copper in silver, and the other a saturated solid solution of silver in copper. Hence, we have two, instead of one, saturated solid solutions crystallizing side by side, and the lowest conductivity is afforded by them just as an alloy of the composition Au-Ag gives the lowest conductivity of all gold-silver alloys. Any addition of silver or copper to Levöl's alloy is equivalent to diluting one or other of the solid solutions of which it consists, and the conductivity is improved thereby.

If the conductivity of a series of steels containing practically a constant percentage of manganese, but varying in carbon, be measured in both the hardened and annealed conditions, and the results plotted, it will be found that the quenched steels not only have a higher resistance, but that the curves diverge from each other directly in proportion to the carbon content. The hardening of steel changes the carbon from a segregated to a dissolved condition. This explains the divergence of the curves, for the increasing amount of dissolved carbon increasingly augments the re-

sistance. If, however, steels of constant carbon and varying manganese be similarly treated, the curve of the quenched specimens will fall below that of the annealed specimens, but will be practically parallel to it.

The difference between the two curves in this case is due to the retention of all the carbon in the dissolved state, but the effect due to increasing manganese is the same in both conditions, for it crystallizes isomorphously with the iron, and—for low percentages—is contained in the iron crystals, whether they are quenched or not.

From what has preceded, it will be seen that although electrolytic and metallic conductivity are so different in nature, yet the question of solubility seems to affect or control metallic conduction, and there exists an atomic relation between the effect of different dissolved substances upon the conductivity of the principal metal or solvent. All the recent work upon alloys tends to strengthen the idea that alloys in many of their properties resemble liquid solutions, and with a brief summary of these points of resemblance we will close. Graham and Roberts-Austen have shown that at temperatures much below their melting points metals diffuse into one another, like a salt into water. Like liquids, metals may or may not be perfectly miscible. In the latter case, Dr. Alder Wright has shown that a fused mass of two immiscible metals on cooling separates into layers, and that the layers do not consist of pure metals, but that each metal retains a little of the other in solid solution.

In general, solubility increases with the temperature. Professor Spring and others have shown that metals will flow under pressure, and that by the same means, mixed metals may or may not react chemically when thus brought into intimate association. Molecular mobility increases with the temperature in metals as well as in liquids. Upon cooling molten binary alloys, the phenomena attending solidification are strongly suggestive of the behavior of freezing salt solutions. It has been shown by Heycock and Neville that the depression of the freezing point of one metal by the addition of another to it follows the laws of Coppet and Raoult. The eutectic alloy of Guthrie is the analogue of the "cryohydrates" of physical chemistry, and Roozeboom, Le Châtelier and others have shown that the phase rule of Gibb applies quite as well to the explanation of questions of equilibrium in



alloys as it does to the explanation of similar problems in regard to liquids and gaseous mixtures.

In view of all this evidence, it may not be surprising, but it is nevertheless a very interesting coincidence, that almost simultaneously three workers of different nationalities in three different countries should have observed independently another confirmation of the periodic relation of the elements and of the general applicability of the solution theory to the explanation of the properties of alloys.

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## ABSTRACTS

(From recent articles on Metallography and related subjects.)

**The Heat Treatment of Steel under Conditions of Steel Works Practice.** Alfred Campion. *Journal Iron and Steel Institute*, 1903, Vol. I. — Mr. Campion, Carnegie Research Scholar, presented an exhaustive paper bearing the above title at the May, 1903, meeting of the Iron and Steel Institute. The paper includes numerous tables showing results of physical tests and many photomicrographs exhibiting the structure of the samples treated.

The author during the time which he was connected with works frequently observed that the results obtained on such small samples were very different to those arrived at in actual practice when large masses of steel were subjected to similar heat-treatment. He therefore decided to investigate the heat-treatment of steel under works conditions, in the hope that the results of such an investigation would be of some value to those engaged in the manufacture and manipulation of the metal.

With this object in view a number of round bars, varying in size from 1½-inch to 6-inch diameter, and in carbon content from 0.20 to 0.65 per cent, have been heated to different temperatures for various lengths of time. The bars after treatment have been subjected to as complete a set of mechanical and other tests as possible, to determine the influence of the particular treatment on the strength, ductility, and general physical properties.

The heatings have been carried out mostly in a gas-fired reheating furnace, but some of the smaller bars were heated in a direct coal-fired furnace.

The effect of the heating has been determined by submitting the bars to as complete a series of such mechanical tests as can be carried out in any well-appointed works, and with which all practical men are familiar and can readily interpret.

The practical results obtained under the drop test, and in the testing machine, have been augmented by microscopical examinations and other tests, which are fully described in the paper.

The scheme of work planned for the research was to treat round bars  $1\frac{1}{2}$ -inch, 4-inch and 6-inch diameter, of four steels containing 0.20, 0.35, 0.50 and 0.65 per cent of carbon, at a number of different temperatures and lengths of time, and afterwards test them in various ways, as shown below. Duplicate experiments have been made for each temperature as far as it was possible to do so.

It was originally intended to have had all three sizes of bars of rolled material, but as it was not possible to obtain the 6-inch diameter bars of rolled material, they were forged from 8-inch by 8-inch blooms. The bars were  $4\frac{1}{2}$  feet long, weighing just under 4 cwts. each. The 4-inch diameter bars were also  $4\frac{1}{2}$  feet long, weighing about  $1\frac{3}{4}$  cwt. each. The  $1\frac{1}{2}$ -inch round bars were 2 feet long, and weighed about 14 lbs.

The bars after being subjected to the heat-treatment were tested and examined in various ways, as shown below.

#### $1\frac{1}{2}$ -INCH DIAMETER ROUNDS

(1) Cooling curves taken and critical points determined in each steel.

(2) Complete chemical analysis of drillings taken from several bars of each steel.

(3) Bending tests.

(4) Tensile tests — elastic and ultimate stress, percentage of elongation and contraction of area at fracture.

(5) Photomicrograph of average structure of a transverse section of each bar.

#### 4-INCH AND 6-INCH DIAMETER ROUNDS

(1) Cooling curves and critical points determined for each steel.

(2) Carbon determined by combustion in every bar.

(3) Complete chemical analysis of half the bars.



- (4) Drop tested each bar where possible to destruction.
- (5) Two tensile tests for each bar, one cut from the centre and one from the outside. See Fig. 1.
- (6) Two compression tests from one bar of each treatment, cut from centre and outside of bars. See Fig. 2.
- (7) Three photomicrographs from each bar, representing the structure on the outside, the centre, and the portion half-way between the centre and outside of the bars.
- (8) Electrical conductivity of strips cut through the diameter of the 6-inch bars.

In carrying out the heatings the temperatures were controlled by a portable form of the Le Chatelier pyrometer. Several pyrometers were in use in order to make sure that all the bars in the furnace were at as near as possible the same temperature.

In taking the temperature of the bars it was necessary to take precautions for preventing the flame playing directly upon the thermo-couple, and the following simple plan was adopted: The bars were covered with a piece of large angle or channel, and the couple was put under the covering and jammed close against the bars. This method of shielding the junction from direct action of the flames proved very convenient, as the piece of angle could easily be shifted to any part of the bar at which it was desired to take the temperature.

The bars were frequently rolled round during the heating to ensure them being equally heated all through.

The micro-sections examined in the course of the investigations have all been prepared in as nearly as possible the same manner, so as to have the structures developed under similar conditions.

The method of etching adopted, and which has given excellent results, was to immerse the section for exactly thirty seconds in alcohol containing 1 per cent of nitric acid. After washing and drying the etched surface was lightly rubbed with a soft cloth moistened with benzene.

#### CONSIDERATION OF THE RESULTS OF THE VARIOUS TESTS

*Bending Tests.*—In the case of the 0.20 per cent carbon steel, very little difference in properties of the 1½-inch bars is made apparent by this method of testing. The steel containing 0.35 per cent of carbon shows somewhat greater variations in the bending

angles than was the case with the 0.200 per cent carbon steel. The steels containing 0.500 per cent and 0.700 per cent carbon show very considerable difference by this method of testing.

*Falling Weight Tests.* — This is by far the most important of the tests dealt with in the present paper. It is, in fact, the only means of determining the shock-resisting power of the steel.

In the case of the steel containing 0.20 per cent carbon the 6-inch forged bar is very much stronger than the 4-inch rolled bar, the maximum resistance to sudden shock of the latter being only about 58 per cent of that given by the 6-inch forged bar. The forged bar reaches its maximum strength when treated at  $850^{\circ}\text{C.}$ , as against  $760^{\circ}\text{C.}$  for the 4-inch bar. It is difficult to say how much of this difference in resistance to shock is due to the mass, as the 4-inch bar was rolled whilst the 6-inch bar was forged. This remark applies to all the four steels used in the investigation.

Passing to the steel containing 0.35 per cent carbon, the forged bar is still much superior to the rolled bar, but not to so large an extent as in the softer steel. The rolled bar has a maximum resistance of 66 per cent of that possessed by the forged bar. Both bars in this case give the best results at about  $750^{\circ}\text{C.}$ , although the falling off takes place at  $100^{\circ}$  higher in the rolled bar than in the forged. The total foot-pounds of energy to produce fracture is 200,000 less than required by the 0.2 per cent carbon steel. The steel containing 0.55 per cent carbon has maximum strength of only half that of the 0.20 per cent steel under this test, and the forged and rolled bars are about equal in this respect. The temperature giving highest result for the rolled bar is within fifty degrees of that at which the forged bar gives the best test. Passing to the next steel, containing about 0.65 per cent carbon, it is interesting to notice that the rolled bar possesses considerably greater strength than the forged bar, the latter having only about 75 per cent the strength of the rolled material. The rolled bar also reaches its maximum at a lower temperature than does the forged bar.

*Tensile Tests.* — As has already been pointed out, the results obtained by subjecting the material to a steady pull in the testing machine do not show any very pronounced differences. They do not therefore give at first sight any indication of the shock-resisting qualities of the steel.



The percentages of contraction of area and elongation appear to vary within very narrow limits.

Kirkaldy\* concludes, as the results of experiments, that the contraction per cent where rupture occurs in the usual form of test piece affords information as to the value of steel. The author has endeavored to trace some relation between the contraction at fractures, and a comparison of the contraction of area curves with those of bending test results show that there is a very good agreement between the two results. In the case of the 0.65 per cent carbon steel, the agreement is less marked owing to the small percentages of contraction of area of such material.

A reference to the figures shows that there exists a fairly close agreement between the results of the two methods of testing in the case of steel containing 0.200 per cent of carbon; but in the case of the steels containing 0.35 and 0.55 per cent of carbon, the percentages of contraction of area at fracture do not appear to give any indication as to the behavior of the material under sudden shock.

Professor Åkerman,† of Stockholm, takes the fraction formed by dividing the limit of elasticity by the breaking load as a measure of the sample to resist impact.

It is to be observed that Åkerman's criterion for resistance to impact is also a criterion for ductility, for the smaller the ratio of the elastic to ultimate stress, the greater the force beyond the limit of elasticity that the material can resist without fracture, and the tougher accordingly it is.

The author has been unable to trace any relation between Åkerman's criterion and the bending angles of the 1½-inch round bars. It was found that no agreement exists between the ratio of elastic to ultimate stress and the resistance to impact in the case of the 0.200 per cent carbon steel. In the case of the other steels, the smallest ratio of elastic to ultimate stress is obtained in the bar giving the highest drop-test result.

It thus appears that for soft steel containing about 0.20 per cent of carbon, the percentage of contraction of area at fracture of the usual test bars affords an indication of the resistance to impact; whilst for steel containing from 0.35 to 0.70 per cent

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\* "Experiments on Wrought Iron and Steel," by David Kirkaldy.

† *Journal of the Iron and Steel Institute*, No. II, 1879.

of carbon, the ratio of elastic to ultimate stress affords similar information.

*Electrical Conductivity Tests.*—The author had hoped to have made a complete series of experiments in this direction on strip cut out both longitudinally and transversely from the bars. Time, however, did not permit of this being accomplished.

Some of the transverse tests have been made, but so far the author has been unable to trace any relationship either with the structure or mechanical tests.

The general conclusions to be drawn from the results obtained may be briefly summarized as follows:

Small sections of rolled steel containing 0.20 per cent carbon can be heated between very wide limits of temperature without seriously impairing its power of resisting sudden shocks, as shown by results of bending tests. The results of experiments given above indicate, however, that  $850^{\circ}\text{C.}$  is the maximum temperature to which such material should be heated.

The rate of cooling to  $\text{Ar}_1$  appears to have considerable influence upon the result; in the case of material of this description heated to  $800^{\circ}\text{C.}$  and higher, slow cooling tends to produce brittleness, a fact which probably explains some of those abnormal results often met with in works. In rolling small sections it is the practice in many works to stack the bars on the bank whilst still hot; this means that they are either cooled very slowly through  $\text{Ar}_1$ , or are kept for a very long time between  $500^{\circ}$  and  $600^{\circ}\text{C.}$  The experiments described in the paper point to this as a dangerous temperature at which to treat the material.

A case of some angles being rendered brittle in this manner recently came under the author's notice. Heating the material to about  $800^{\circ}\text{C.}$  and allowing to cool fairly rapidly entirely removed the brittleness.

The results obtained upon larger sections of rolled steel containing 0.200 per cent carbon, indicate  $800^{\circ}\text{C.}$  as the maximum temperature at which such material should be heated to obtain it in the most suitable condition to resist sudden shock. This temperature is slightly lower than the maximum obtained for the smaller bars.

Passing to the experiments on forged bars of this steel and of large mass, the results point to  $700^{\circ}$  to  $850^{\circ}\text{C.}$  as the limits of temperature at which such material should be treated to obtain



the maximum resistance to sudden shock. It appears that at temperatures between  $500^{\circ}$  to  $650^{\circ}$  C. brittleness is produced in a very marked degree, especially if followed by slow cooling.

The second set of experiments described upon this size of bar, show very clearly the effect of heating to temperatures between  $500^{\circ}$  and  $1000^{\circ}$  C., and the results point to  $800^{\circ}$  C. as the best temperature at which to treat such material. The results of this set of experiments are of additional interest. The author being unable to spare the necessary time to go to the works and superintend the heatings in this case, requested his friend, Mr. Farquharson, the chemist at Hallside Works, to treat the bars according to his instructions, and afterwards test them under the falling weight. The results absolutely confirm those obtained by the author himself, and also prove the value of a pyrometer in controlling the heatings in ordinary works practice.

Steel containing about 0.30 or 0.40 per cent of carbon in small rolled sections appears to be improved as regards its shock-resisting qualities when heated to temperatures varying from  $650^{\circ}$  to  $800^{\circ}$  C., whether followed by slow or rapid cooling.

Heating between  $500^{\circ}$  and  $600^{\circ}$  C. does not produce the brittleness which occurs in 0.20 carbon steel under similar conditions.

Large rolled bars of this give the best results after heating to temperatures of  $700^{\circ}$  to  $800^{\circ}$  C. with the maximum resistance at  $760^{\circ}$  C. When heated at  $900^{\circ}$  C. and higher, a pronounced brittleness develops. Slow cooling does not seem to be very injurious.

Forged bars 6 inches in diameter of this steel appear to be brought into the best condition for resisting sudden shock by heating to temperatures between  $700^{\circ}$  to  $760^{\circ}$  C., followed either by slow or rapid cooling. The bar heated to  $800^{\circ}$  C. shows a marked falling off in the drop-test result, but as only one experiment was made at this temperature, too much reliance must not be placed upon the result, which might be due to accidental causes.

Steel containing 0.50 to 0.60 per cent carbon in small rolled sections appears to be in its toughest and strongest condition after being heated to  $700^{\circ}$  to  $800^{\circ}$  C., and followed by either slow or rapid cooling. The larger rolled bars of this steel appear to require the same treatment as the  $1\frac{1}{2}$ -inch bars.

The forged bars require to be heated to a slightly lower temperature than the rolled bars in order that they may be in the

toughest condition. The steel containing between 0.60 and 0.70 per cent of carbon in small rolled sections as used in the author's experiments possess greatest ductility and resistance to sudden shock, as determined by bend test, after being heated to  $600^{\circ}$  to  $700^{\circ}$  C. The 4-inch rolled bars of this steel give the best drop-test results at about  $720^{\circ}$  C. The rate of cooling apparently does not very much influence the results.

This steel in the forged bars of 6-inch diameter gives the best result under the falling weight test after being heated to  $700^{\circ}$  to  $760^{\circ}$  C., and followed by either rapid or slow cooling. Heating this grade of steel in large bars to temperatures of over  $850^{\circ}$  C. produces brittleness, which is very marked if the temperature exceeds  $900^{\circ}$  C.

**Determination of the Points of Allotropic Changes of Iron and its Alloys by the Measurement of the Variations in the Electric Resistance.** O. Boudouard. *Journal Iron and Steel Institute*, 1903, Vol. I.—At the May meeting of the Iron and Steel Institute, Mr. Boudouard, Carnegie Research Scholar, presented an exhaustive paper bearing the above title.

The work was carried out at the College of France, in the laboratory of Professor Le Chatelier.

The paper is divided into sub-sections as follows:

I. Historical review.

II. Plan of experiments. Measurement of the resistances. Description of the apparatus. Method of heating. Measurement of the temperatures. Conduct of an experiment.

III. Results of experiments. Tables and curves.

IV. Conclusions.

In two appendices the thermo-electricity of iron and steel, and the nature of the gases occluded in these metals, are dealt with.

Among the physical properties of iron and steel, the variations of which are characteristic of the points of transformation in these metals, those of expansion and electric resistance form perhaps the most instructive object of study. The measurement of these can in fact be effected at constant temperature, thus helping to shorten the delays at the points of transformation, the presence of which introduces confusion into the interpretation of the results obtained by the observation of the laws of the heating and cooling



of steels. The difficulties involved in making the experiments are perhaps greater, but the trustworthiness of the conclusions to be drawn compensates largely for the additional trouble.

Numerous experiments in the measurement of the expansion of metals have already been carried out by Svedelius, Le Chatelier, Charpy and Grenet. But an investigation of the electric resistances at high temperatures has never been undertaken in a comprehensive manner. The experiments of Hopkinson and Le Chatelier, the only ones hitherto made, were conducted with metals of unknown composition.

It is now many years since the first investigations relating to the electric resistance of iron and steel were published. In 1873, Benoit gave as the specific resistance of annealed steel, 10.99 microhms, and of annealed iron, 12.16 microhms. The further researches of Matthiessen and Vogt, of Schneider and Company, of Martens, Paalzow, Wedding, Le Chatelier, and Barrett defined the influence of the carbon, the manganese, and the silicon, the effect of the carbon being clearly less marked than that of the manganese.

Many scientists have turned their attention to the study of the electric conductivity of iron and steel in functions of the temperature, and have attempted to represent the law of variation, either by means of linear equations or of parabolic equations.

According to Harrison the curve of resistance for iron does not change its parabolic form until nearly 800°. It then becomes linear and remains so within the limits to which the experiment can be carried. On taking the equation of resistances as  $r_t = r_0 (1 + at)$ , Barus and Strouhal found that  $a$  diminished as  $r$  increased. This formula holds good from +10° to +35°. Beyond this point, however, parabolic equations must be used, the application of which is greatly limited on account of the chemical or molecular changes which the iron undergoes.

The electric resistance of 30 to 37 per cent nickel steels has been studied by C. E. Guillaume. The specific resistance, which is high and varies little from one alloy to another, lies between 80 and 90 centimeter-microhms. The electric resistance varies regularly with the temperature, even during the magnetic change. Between 20° and 150° it may be represented by a function of the second degree of which the second term is very small. The less expansible alloys vary more than those with a higher capacity for

expansion. Annealing diminishes the resistance slightly. Thus in the case of nickel steels new evidence is afforded that the variation of the electric resistance with the temperature cannot be considered as simply due to the expansion.

The electric resistance is similarly influenced by the presence of foreign substances which are to be met with in iron and steel. The influence of carbon, silicon, manganese, nickel, etc., has accordingly been determined by Le Chatelier, and since his results relate to the same specimens of metal which have been used by the author in the experiments about to be described, a short summary of his determinations is given below:

### *Influence of the Carbon*

Composition			Specific Resistance in Centimeter-Microhms
C.	Mn.	Si.	
0.06	0.13	0.05	10.0
0.20	0.15	0.08	12.5
0.49	0.24	0.05	14.0
0.84	0.24	0.13	16.0
1.21	0.21	0.11	18.0
1.40	0.14	0.09	18.4
1.61	0.13	0.08	19.4

The electric resistance thus increases with the carbon content, the average increment being 7 microhms for each increase of 1 per cent of the carbon by weight, or 1.5 microhms for each increase of one atom of carbon in 100 atoms of iron and carbon.

### *Influence of the Silicon.*

Composition		Specific Resistance in Centimeter-Microhms
C.	Si.	
0.2	0.1	12.5
0.2	2.6	38.5
0.8	0.1	15.8
0.8	0.7	26.5
0.8	1.3	33.5
1.0	0.1	17.8
1.0	0.6	25.5
1.0	1.1	32.0



By varying the silicon content the increase in the resistance is found to be 14 microhms for each increase of 1 per cent of the silicon by weight, or 7 microhms for an increase of one atom of silicon in 100 atoms of the alloy. The effect of silicon in raising the resistance is therefore much greater than that of carbon.

### *Influence of the Manganese*

Composition			Specific Resistance in Centimeter-Microhms
C.	Mn.	Si.	
0.9	0.24	0.1	17.8
0.9	0.95	0.1	22.0
1.2	0.83	0.2	24.5
1.2	1.8	0.9	40.0
1.0	13.0	0.3	{ 66.0*
			{ 80.0†

\* Magnetic.

† Non-magnetic.

The increase is about 5 centimeter-microhms for each rise of 1 per cent in weight or in atoms of manganese.

In the case of nickel the increase in resistance, with reference to the calculation of an alloy containing 1 per cent of nickel, varies widely with the proportion of nickel. For alloys with a total nickel percentage below 5 per cent, the increase has been found to vary from 3 to 7 microhms for every additional 1 per cent of nickel.

The influence of chromium, of tungsten, and of molybdenum in raising the resistance is very small. This points to the conclusion that these metals are isolated within the alloy in a state of definite combination. This view agrees also with the results formerly obtained by Carnot in his chemical research upon steels of the same character.

The most recent investigations of the influence of foreign substances in iron upon the electric resistance of the metal are those of Benedicks on the one hand and of Barrett, Brown and Hadfield on the other.

The following equation has even been drawn up by Benedicks, which enables the calculation to be made of the resistance of steel at the ordinary temperature:

$$R = 7.6 + 26.8 \Sigma C.$$

$\Sigma C$  represents the percentage of the sum of the hardening carbon and of the other elements in solution, expressed in terms of the carbon. That is to say —

$$\Sigma C = \text{hardening carbon} + \frac{Z \times 12}{28.4} (\text{Si}) + \frac{Y \times 12}{55} (\text{Mn}) + \dots$$

The resistance of pure iron is therefore 7.6 centimeter-microhms.

Quenching only affects the electric resistance of steel when it is performed at a temperature above that of the hardening temperature ( $700^{\circ}$ ). The resistance then continues to increase as the temperature at which quenching takes place is raised, the value attained being proportionately higher, the richer the steel is in carbon. The increase in resistance of iron due to the presence of hardening carbon averages 45 microhms for each additional 1 per cent of carbon by weight, or 7 microhms for an increase of one atom of carbon in 100 atoms of the alloy.

At high temperatures chromium accentuates still more the increase in resistance which quenching would have produced in steel containing only carbon. Tungsten, on the other hand, diminishes the resistance. This supports the conclusion that chromium, a metal analogous to iron, after quenching, assumes at least in part the form of an isomorphous mixture, as do the nickel and manganese. The tungsten, however, appears both before and after quenching to remain isolated in the mass.

The most important publications bearing upon the particular point of view of the allotropic changes now under consideration are those of Le Chatelier and of Hopkinson.

Hopkinson gives the following results: For soft iron wire, the temperature at which a change occurs is  $855^{\circ}$ , and for a piano wire containing 0.724 per cent of carbon the temperature is  $812^{\circ}$ . For manganese steel the coefficient of temperature remains constant up to  $1000^{\circ}$ . For steel containing 25 per cent of nickel the change, which occurs during heating to redness, is not reversed except at a temperature below  $0^{\circ}$ . This alloy is therefore able to exist throughout a wide range of temperatures in two different states, one of which is magnetic and the other non-magnetic.

The following conclusions were arrived at by Le Chatelier: First, in the case of iron, the change at  $850^{\circ}$ , which is difficult to detect by means of the calorimetric method, is very plainly indicated by the electric method. This change would also occur in steel in the same manner as in soft iron. On the other hand,



the change at  $750^{\circ}$ , and that at the point of recalescence, produce a scarcely noticeable effect upon the electric resistance. Secondly, in the case of nickel steels, the displacement of the points of transformation varies continuously with the composition of the alloy, and the change is not divided into two stages. The nickel-iron alloys are not constituted by a combination of the component metals, effected by bringing them in contact until one or other is in excess of the required proportion, but they form a chemically homogeneous and isomorphous mixture. Thirdly, the investigation of the electric resistances has enabled certain allotropic changes to be characterized which were hitherto unknown. Particularly those at  $730^{\circ}$  in brass, at  $500^{\circ}$  in bronze and aluminium, at  $350^{\circ}$  in zinc, and at  $690^{\circ}$  in a copper-iron-nickel alloy.

Acting on the advice of Professor Le Chatelier, the author has therefore undertaken a systematic study of the variations of the electric resistance of iron and steel in functions of the temperature.

In pursuing this object he has availed himself of the specimens which were used by Madame Sklodowska Curie in investigating the magnetic properties of steels, and by Professor Le Chatelier himself for his researches upon the electric resistance of steels. These specimens of steel are in the form of square bars, the side of which measures 1 centimeter, the length being 20 centimeters. Some of the bars are round, with a diameter of one centimeter. Their chemical composition was determined by Messrs. Moutonnet and Goutal in the laboratory of the Paris School of Mines. It is preferable to use specimens of a large section rather than simple wires, for the reason that the homogeneity of the metal may be better depended upon to remain constant during a series of experiments than would be the case if wires of only one-half or of one millimeter in diameter were used. If, for instance, in the case of steel, the conditions of test render decarburization possible, the change in the chemical composition would extend throughout the whole mass in a wire of small diameter, whereas in a bar of large section the effect would be negligible since it would be limited only to a very thin layer on the surface.

From his exhaustive and carefully conducted experiments, Boudouard arrived at the following conclusions:

The combined results obtained from these experiments estab-

lish the perfect reversibility of the phenomena of the electric resistance of iron and its alloys in functions of the temperature, at least for those temperatures situated outside of the zones in which the allotropic changes occur. In the neighborhood of the critical points the deviation between the heating curves and the cooling curves, in the case of the carbon steels, is greater the higher the proportion of carbon. The steels containing foreign metals, such as chromium, manganese, or tungsten, present in general distinctive features of the same type as those noted in the case of steels containing above 1 per cent of carbon. This conclusion might have been anticipated. For since chemical reactions or the allotropic changes proceed with a certain amount of speed only while the system is still far removed from the conditions of equilibrium, it is probable that on nearing the point of transformation the changes proceed with extreme slowness. In the particular case of iron, the changes will be still further retarded by the solid state of the mass, and by the presence of carbon and of foreign metals.

The curve of the variations of electric resistance of irons and steels does not alter its parabolic form between the ordinary temperature and that at which the transformations begin to occur. But from 800° onwards they become linear. The following equations plainly represent the phenomenon above the temperature of 800°:—

Steel with 0.205 per cent carbon	. . . . .	R = 42.8 + 0.0486 t
“ “ 0.493 “ “	. . . . .	R = 47.0 + 0.043 t
“ “ 0.841 “ “	. . . . .	R = 72.7 + 0.023 t
“ “ 0.82 “ “	. . . . .	R = 92.9 + 0.028 t
“ “ 1.05 “ “	. . . . .	R = 92.0 + 0.029 t
“ “ 1.16 “ “	. . . . .	R = 90.6 + 0.0283 t
“ “ 1.38 “ “	. . . . .	R = 97.75 + 0.0301 t
Chrome steel, C 1	. . . . .	R = 74.2 + 0.031 t
“ “ C 2	. . . . .	R = 67.55 + 0.017 t
“ “ C 3	. . . . .	R = 67.3 + 0.0255 t
Tungsten steel, V 1	. . . . .	R = 67.8 + 0.015 t
“ “ V 2	. . . . .	R = 59.0 + 0.032 t
“ “ V 3	. . . . .	R = 66.4 + 0.0183 t
Manganese steel, M 2	. . . . .	R = 92.8 + 0.0226 t
“ “ M 3	. . . . .	R = 84.9 + 0.033 t
Nickel steel, N 1	. . . . .	R = 116.7 + 0.045 t
“ “ N 2	. . . . .	R = 125.7 + 0.0305 t
“ “ N 3	. . . . .	R = 118.0 + 0.047 t



In the case of the specimen of iron investigated by the author, the three following linear equations represent the phenomenon:

$$\begin{array}{llll} \text{From } 655^{\circ} \text{ to } 775^{\circ} & . & . & . & R = -51.9 + 0.189 t \\ \text{" } 781^{\circ} \text{ " } 877^{\circ} & . & . & . & R = 39.3 + 0.071 t \\ \text{" } 896^{\circ} \text{ " } 1170^{\circ} & . & . & . & R = 74.4 + 0.031 t \end{array}$$

In the case of carbon steels the electric resistance increases with the carbon content. The addition of chromium and of tungsten increases the resistance sensibly in the ratio of 1 to 2. But this ratio of increase is not maintained at high temperatures, at which point the chrome and tungsten steels have a degree of resistance about equal to that of carbon steels. Manganese at ordinary temperatures causes the resistance to become at least two or three times greater. Nickel produces a much greater increase in the resistance than manganese. In the case of both of these metals the ratio of increase is much less in the hot state than in the cold state.

## I. CARBON STEELS

The transformations  $A_1$ , the point of recalescence, and  $A_2$ , the point of loss of the magnetic properties, in low carbon steels have little effect upon the electric resistance. Their rate of progress is clearly defined, and the range of temperature is fairly wide. The transformation  $A_3$ , on the contrary, is sudden. The electric resistance, after rising considerably above that of the metal at ordinary temperature (it is 8 times higher in the case of the carbon steels, 4 or 5 times higher in the case of chrome and tungsten steels, and 3 times higher in the case of manganese and nickel steels), does not show a sensible increase above this temperature. This point of transformation is even noticeable in steels containing 0.5 per cent of carbon, the temperature at which it would occur being in the neighborhood of  $800^{\circ}$ . The existence of the point  $A_3$  in these steels is, however, not established with certainty.

As the carbon content gradually increases, the points  $A_1$ ,  $A_2$ ,  $A_3$ , as has already been observed, converge, until finally they coincide. The phenomenon of the allotropic change then becomes very strongly marked.

On examining the curves appended to this paper, it will be

seen that the points of the allotropic changes in the carbon steels investigated may be fixed as follows:

	A <sub>3</sub>	A <sub>2</sub>	A <sub>1</sub>
	Degs.	Degs.	Degs.
Commercial steel . . . . .	885	775	—
Steel with 0.205 carbon } in heating . . . . .	835	780	710
} in cooling . . . . .	835	—	710
Steel with 0.493 carbon } in heating . . . . .	825(?)	792-768	—
} in cooling . . . . .	—	734-722	700
} mean . . . . .	825(?)	755	700
Steel with 0.841 carbon } in heating . . . . .	670-722-728-770		
} in cooling . . . . .	682		

In the case of the last mentioned steel the points A<sub>1</sub>, A<sub>2</sub>, A<sub>3</sub> converge in a single point situated, according to the mean curve, at the temperature of 700°.

The heating curve, in the case of steel containing 0.841 carbon, presents a peculiar form. After a change of curvature at 670°, it reaches a maximum point at 722°, then falls to a minimum at 728°, and becomes almost a straight line after 770°. It will be noted that the portions of the curve below 722° and beyond 728° almost form a prolongation one of the other, and by joining direct the points 716°, 722°, 728° another mean curve can be constructed which brings out two remarkable points, the one at 696° and the other at 745°. The points 696° and 700° are evidently identical.

With the high carbon steels (0.82 to 1.38 per cent carbon), during the period of heating up, the pyrometer indicates very plainly a diminution in the rate of increase of temperature, and sometimes a pause. During cooling a similar diminution in the rate of falling is to be observed, then at a given moment a very clearly defined pause, the duration of which reaches seven or eight minutes.

The temperature of the point of transformation (the point of convergence of A<sub>1</sub>, A<sub>2</sub>, A<sub>3</sub>) in steels containing from 0.82 per cent carbon and upwards rises progressively from 700° to 725°; that is to say, it rises above a minimum point corresponding to eutectic steel.



	0.82 per cent Carbon Steel	1.06 per cent Carbon Steel	1.15 per cent Carbon Steel	1.38 per cent Carbon Steel
Point of transformation during heating	Degs. 728-760	Degs. 730-760	Degs. 739	Degs. 750
Point of transformation during cooling	680-667	695-691	691	700
Mean . . . . .	700-710*	715-720	715	725

\* This result is in absolute agreement with that obtained for steel with 0.841 per cent carbon in the preceding series of carbon steels.

For all these steels the outline of the curves is approximately the same; it may be noted, however, that during heating in the case of the 0.82 and 1.06 per cent steels that the electric resistance increases gradually throughout an interval of 30°. In case of 1.15 and 1.38 per cent carbon steels, on the other hand, nearly the whole of the increase in resistance occurs at constant temperatures. The same observation applies to the cooling curves.

## 2. CHROME STEELS

The interpretation of these curves presents some difficulties, since the evidence seems to support the theory of two points of transformation:

		Degs.	Degs.
C 1	in heating . . . . .	760-782-788-792	—
	in cooling . . . . .	—	716
C 2	in heating . . . . .	792-809	—
	in cooling . . . . .	—	719
C 3	in heating . . . . .	809	—
	in cooling . . . . .	—	719

Although during the course of the investigation of chrome steels, the author noted some clearly defined arrest points in the variation of temperature of the bar under experiment at 790° during heating, but principally at 716°-719° during cooling, yet he is of opinion that these points do not correspond to the transformation points, properly so-called. They indicate that the phenomenon of molecular transformation had at that moment reached the point of maximum intensity, though it ought strictly speaking to have begun at a lower temperature in the heating curve or at a higher temperature in the cooling curve, since the

two temperatures should coincide. On comparing the heating curves and cooling curves,  $\delta$ , of the chrome steels, their outline indicates clearly the existence of an anomaly at the same temperature. It may be assumed, therefore, that there is only a single point of transformation in the chrome steels which have been examined occurring at the following temperatures:

	Degs.
C 1 . . . . .	765
C 2 . . . . .	750
C 3 . . . . .	750

### 3. TUNGSTEN STEELS

The points of transformation were found to occur at the following temperatures:

	Degs.	Degs.	Degs.
V 1 { in heating . . . . .	792 <sup>1</sup>	750-730	—
{ in cooling . . . . .	809-792	—	530-500
{ mean . . . . .	—	730	512
V 2 { in heating . . . . .	—	780-750	—
{ in cooling . . . . .	—	667	532-522
{ mean . . . . .	—	716	540
V 3 { in heating . . . . .	—	780-750	—
{ in cooling . . . . .	—	688	570-550
{ mean . . . . .	—	718	583

With regard to the specimen V 1 there may be some doubt, but for the specimens V 2 and V 3 there is no hesitation in fixing one of the points of transformation at the temperatures 716° and 718°, notwithstanding that very clearly defined arrest points were noted, as in the case of the chrome steels, during the change of temperature of the bar under experiment. The determination of the temperatures of the points of transformation in the tungsten steels V 2 and V 3 confirms the interpretation of the curves for chrome steels which were previously given.

### 4. MANGANESE STEELS

The points of transformation were found to occur as follows:

	Degs.
M 2 { in heating . . . . .	691-728
{ in cooling . . . . .	610-600
{ mean . . . . .	655



During heating the curve becomes distinctly rectilinear from  $950^{\circ}$  onwards. During cooling the curve is rectilinear from  $1170^{\circ}$  to  $770^{\circ}$ . From  $770^{\circ}$  to  $759^{\circ}$  a slight rise in the curve is noticeable. (This anomaly is also reproduced in the curve  $\delta$ .)

				Degs.	Degs.
M 3	{ in heating	.	.	728-750	950
	{ in cooling	.	.	665-658	950
	{ mean	.	.	700	950

Above the temperature of  $700^{\circ}$  both the heating and the cooling curves, as well as the mean curve, show an angular prominence at  $950^{\circ}$ .

## 5. NICKEL STEELS

In the case of the nickel steels the following are the points at which transformation occurred:

				Degs.	Degs.
N 1	{ in heating	.	.	750	—
	{ in cooling	.	.	648	639
	{ mean	.	.	700	660
N 2	{ in heating	.	.	700-710	—
	{ in cooling	.	.	600	571
	{ mean	.	.	655	581
N 3	{ in heating	.	.	710-720	—
	{ in cooling	.	.	610	—
	{ mean	.	.	665	581

The most noteworthy transformation is that which occurs at the respective temperatures of  $700^{\circ}$ ,  $655^{\circ}$ , and  $665^{\circ}$ . It was observed both in heating and in cooling, a circumstance which does not characterize the transformation occurring at a lower temperature. This latter is only noticeable during cooling, but the mean curve undergoes changes of direction at the temperatures of  $660^{\circ}$ ,  $581^{\circ}$ , and  $581^{\circ}$ , sufficiently well marked to enable one to deduce the existence of a molecular change at these temperatures.

*Remarks.* — (1) In the doubtful cases, that is in the case of the chrome and tungsten steels, when there are several temperatures which might be assigned as the point of transformation, it is probable that the matter could be cleared up by resorting to microscopic metallography. For instance, the examination of quenched metals after having been heated to temperatures above

and below the critical points would show the nature of the constituents of these metals. The author much regrets that time did not permit him to undertake a research of this kind. His work would then have been completed by bringing forward experimental proof of a totally different order in support of the conclusions enumerated in Section IV of this paper.

(2) The composition per cent having been given of the special steels investigated in this series of research work, the author has been unable to determine the specific influence of the elements contained in these steels upon the position of the critical points. This influence can only be determined with precision by taking specimens, which with a given percentage of carbon, for instance, contain an increasing proportion of foreign metals (chromium, tungsten, nickel, manganese, etc.). The author hopes shortly to be in possession of three such series of steels containing 0.125, 0.240, and 0.800 per cent of carbon, the other elements being present only as traces. With these it is proposed to undertake experiments with the object of determining the specific influence of each metal upon the allotropic changes of steels.

**Recent Publications.**—*The American Steel Worker*, by F. R. Markham; 343 pages, 8vo. Illustrated. The Derry-Collard Co., 256 Broadway, New York, 1903. Price, \$2.50.—This is essentially a practical book written by a practical man who, to quote his own words, has had "a twenty-five years' experience in the selection, annealing, working, hardening and tempering of various kinds and grades of steel." The author deals exhaustively with the treatment of steel in its various phases, and his book should prove of much value to those engaged in the industrial working of steel. The illustrations are numerous and well prepared. A very good index of 26 pages is appended to the book. The publishers will send the book on approval anywhere in North America.

*Iron, Steel and Other Alloys*, by Henry M. Howe; 457 6×9 in. pages. 120 illustrations. Sauveur and Whiting, Boston. Price, \$5.—In this book which marks an epoch in the literature of Iron, Steel and other Alloys, Professor Howe describes, as he alone could do it, the progress made within the last twenty years



in our knowledge of these metals. The table of contents which is here reproduced in full, shows the character and exhaustiveness of this admirable work. The book also contains a carefully prepared index of 17 pages.

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Special Reference to their Physics and Microstructure,  
their Industrial Treatment and Applications

Edited by ALBERT SAUVEUR

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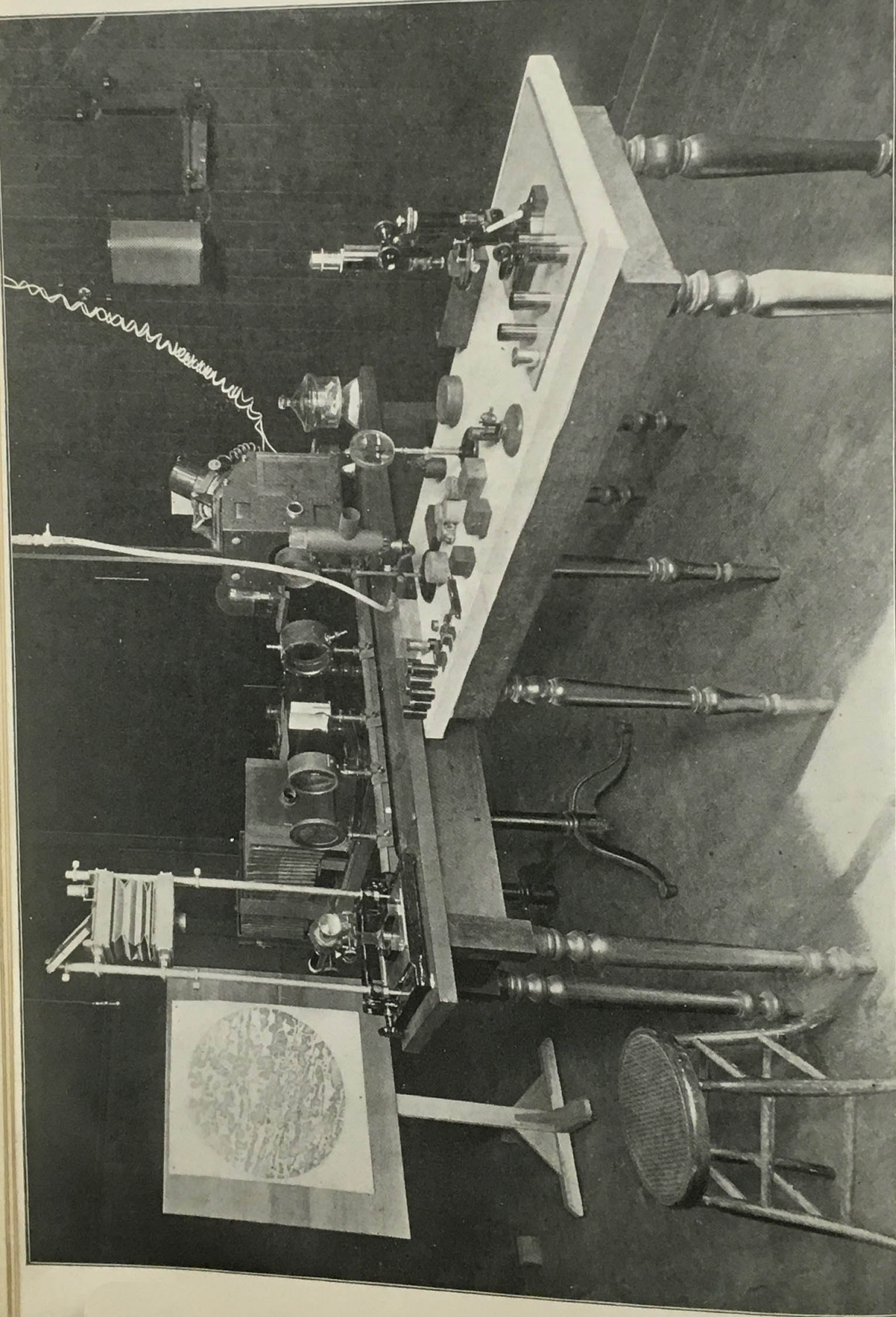
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OUTFIT FOR THE MICROSCOPICAL EXAMINATION OF METALS AND ALLOYS

# The Metallographist

A QUARTERLY PUBLICATION DEVOTED TO THE STUDY OF METALS, WITH  
SPECIAL REFERENCE TO THEIR PHYSICS AND MICROSTRUCTURE,  
THEIR INDUSTRIAL TREATMENT AND APPLICATIONS.

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Vol. V

JANUARY 1902

No. 1

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## OUTFIT FOR THE MICROSCOPICAL EXAMINATION OF METALS AND ALLOYS

THE Frontispiece shows the microscopical,\* photo-micro-graphic and projection equipment of the Boston Testing Laboratories. Every part of this outfit was either especially constructed or selected with the greatest care in view of the special needs of the metal microscopist.

On the light table will be seen a special microscope stand, eye pieces, objectives and other accessories and a Welsbach Lamp and Condenser. A wide range of work may be covered with this simple outfit, the Welsbach Lamp and Condenser yielding a very satisfactory illumination for low and medium high powers. In many instances, it is all that is needed.

On the dark table will be seen the same microscopical outfit together with an illuminating outfit consisting of an arc lamp and a complete optical bench. The optical bench includes two large condensing lenses, one water chamber, two Ray filters, and one large Iris diaphragm. The microscope is placed on the base of a vertical photo-micro camera. On the wall to the right is a rheostat through which the current is made to pass before entering the lamp. This illuminating outfit yields an ideal illumination, of great intensity and steadiness, unobtainable by any other combination. It is the only arrangement by which it is possible to throw the image of the specimen *directly upon a screen*,



a beautiful and instructive display. The structure of the samples may in this way be examined by any number of observers simultaneously, discussed at leisure and with greater profit. The direct projection affords also an excellent means of drawing the structure, greatly superior to the use of the camera lucida.

The image seen on the screen represents the structure of a certain piece of white cast iron as it appears when projected upon it. The original magnification was 500 diameters.

With the assistance of a special polishing machine, it does not require, in the majority of cases, over 10 minutes to polish and otherwise prepare a sample of metal for microscopical examination. The image of the structure may then be thrown upon a screen or photographed if desired. The exposure required with the electric illuminating outfit varies from one to five seconds. By using Carbutt's process plates and drying with alcohol, the negative may be ready for printing within half an hour after the sample has been taken in hand.

With a Welsbach lamp the time of exposure is, of course, considerably longer.

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## THE PROPERTIES OF STEEL CASTINGS \*

By J. O. ARNOLD

THE researches embodied in the papers of which this is the first were commenced about six years ago in the steel-works and laboratories of the Sheffield University College. The plan of campaign was to determine:

1. The influence of chemical composition on the mechanical properties and micro-structures of steel castings.
2. The influence of annealing on the mechanical properties and structures.
3. The mechanical influence of variations in the specific gravities of steel castings.
4. The influence of process, namely, the difference, if any, between crucible and open-hearth castings.
5. The influence of mass—that is to say, the difference between the properties of large and small castings.

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\* Iron and Steel Institute, May 1901.

6. The influence of heat treatment on annealed and unannealed steel castings.

7. The influence of oil quenching on annealed and unannealed castings.

8. The influence of silicon and manganese on the heat of recalescence at the carbon change point,  $A_{r_1}$ , the object of such observations being to obtain, if possible, thermal indications of the formation of double or triple carbides.

9. The relative properties of annealed castings and similar steels after forging.

### CHEMICAL COMPOSITION

In a research designed to ascertain the best standard composition for steel castings, it was, of course, necessary to bear in mind the specifications at present issued by engineers to ensure high-class material. A common specification demands a maximum stress of about 30 tons per square inch—an elongation of 20 per cent on 2 inches, and a bending angle on an inch square bar of at least  $90^\circ$ . In order to ascertain the influence of chemical composition on attaining, excelling, or falling short of the above requirements, it was decided to manufacture series of castings in three distinct chemical groups.

*Group A.*—Nearly pure iron and carbon castings, in which silicon, manganese, sulphur, and phosphorus should be kept low.

*Group B.*—Iron, carbon, and silicon castings, all other elements low.

*Group C.*—Iron, carbon, and manganese castings, low in other elements.

#### *Group A.—Iron and Carbon Castings*

The group dealt with in the present paper is A., consisting practically of iron and carbon. This group, although perhaps the least interesting from a practical works point of view, is really of vital importance, because it forms the base-line from which alone the influence of the elements silicon and manganese can be accurately gauged. This fact does not seem to have appealed to some workers in the field of steel research, and in consequence much acrimonious, but unnecessary, controversy has resulted.



To include all the castings which have been made and tested in Group A. would inordinately lengthen the paper. The results set forth in Table I. must therefore be regarded as merely typical, and the castings therein have been selected to give a fair view not only of the influence of carbon on iron, but also to record a due proportion of those mysterious variations which set at defiance both the skill of the practical man and the science of the theorist. On reference to the table it will be seen that the series consists entirely of crucible steel manufactured from best Swedish iron. It will be shown in Part II. that it is quite unnecessary in general works practice to employ such a costly base. The castings were made in dry composition moulds in the form shown in Fig. 1, namely, a group of four bars about  $1\frac{3}{8}$  inches round, and, of course, tapered to avoid "pulling." From the 3-inch round head four feeders arranged cruciformly lead to the actual bars, the whole group weighing about 28 lbs.

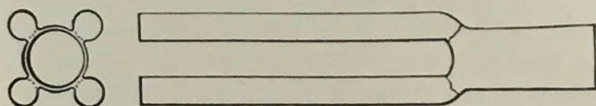


Fig. 1.

Two of the bars were broken off to be annealed, the other two being tested as cast. From such a casting sets of test-pieces containing tensile, compression, bending and specific gravity pieces, together with micro-sections of the material both as cast and after annealing, are readily obtained.

It will be seen that in the seventeen castings selected as types for Group A., the carbon varied from 0.06 to 1.95 per cent. It is hardly necessary to add that only castings above suspicion as regards soundness were included.

#### *Sizes of Test-Pieces*

*Tensile.* — 0.564 inch diameter, or 0.25 square inch area, and 2 inches parallel.

*Compression.* — 0.564 diameter, or 0.25 square inch area, and 2 diameters long.

*Bending.* — 10 inches long and  $\frac{3}{4}$ -inch diameter.

*Micro-sections.* — Transverse only (the material being cast),  $\frac{1}{2}$ -inch diameter by  $\frac{1}{10}$ -inch thick.

*Specific Gravity.* — Polished bars 2 inches long and  $\frac{3}{8}$ -inch diameter.

#### METHOD OF ANNEALING

The bars being somewhat small, it was necessary to protect them from undue scaling. They were therefore annealed in covered cast-iron boxes filled with quicklime. During the annealing process the boxes were maintained at a temperature of about  $950^{\circ}$  C. for about seventy hours. The castings were cool enough for drawing in about another hundred hours. The cooling curve of the furnace is shown in Fig. 2, the co-ordinates being time and temperature. The readings were taken every fifteen minutes by students working in shifts, and the temperature of the furnace during the annealing operation was pyrometrically controlled by experienced students.

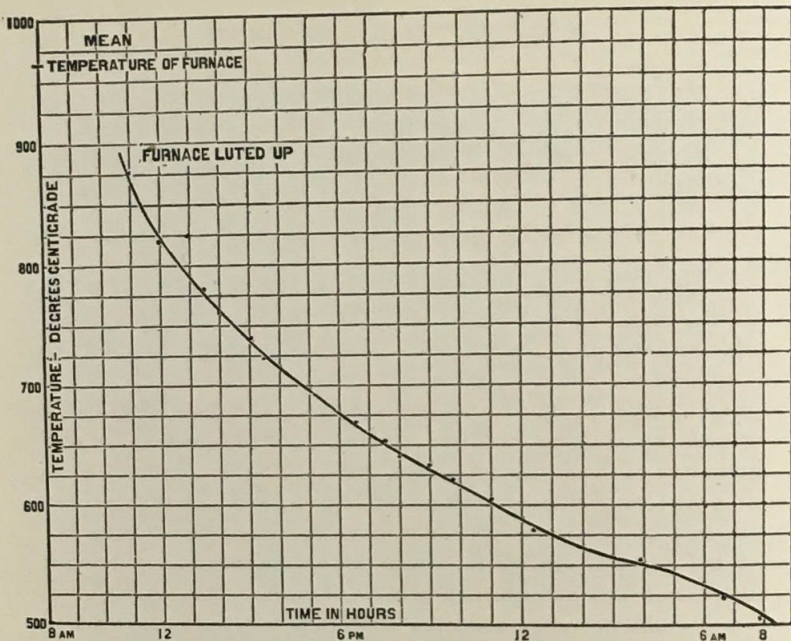
It will be noted that the capability of the constituents to segregate ceases about twenty hours after luting up the furnace; in fact the vital range of temperature in which is determined the ultimate structure of ordinary castings, namely, from  $750^{\circ}$  to  $550^{\circ}$  C. occupies only thirteen hours. The prolonged period of slow cooling from 550 to say  $30^{\circ}$  has little influence on the structure, but is highly necessary from a physical point of view in connection with the question of the unequal contraction of varying masses when cooled too quickly.

In spite of the precautions taken to prevent undue oxidation, it will be seen that during the annealing decided decarbonization has taken place in the supersaturated castings, the figures given being the mean carbon of the tensile test-pieces. These results show how much more readily the carbon in cementite is oxidized than that in martensite.

#### CONSIDERATION OF THE RESULTS—SPECIFIC GRAVITY

The specific gravity results have been quite negative. The only noticeable feature is, that, as a rule, but not invariably, the specific gravity rises slightly on annealing, but speaking broadly no correlation has been established between the densities of the castings and their mechanical properties—a disappointing result,





Cooling Curve of Annealing Furnace. (University College, Sheffield.)

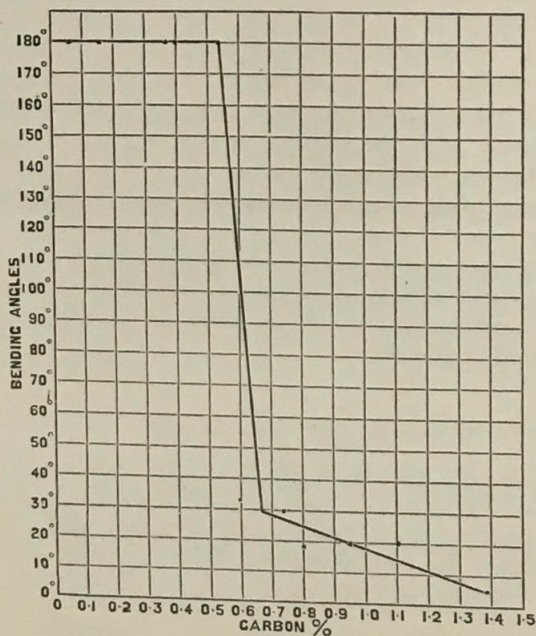
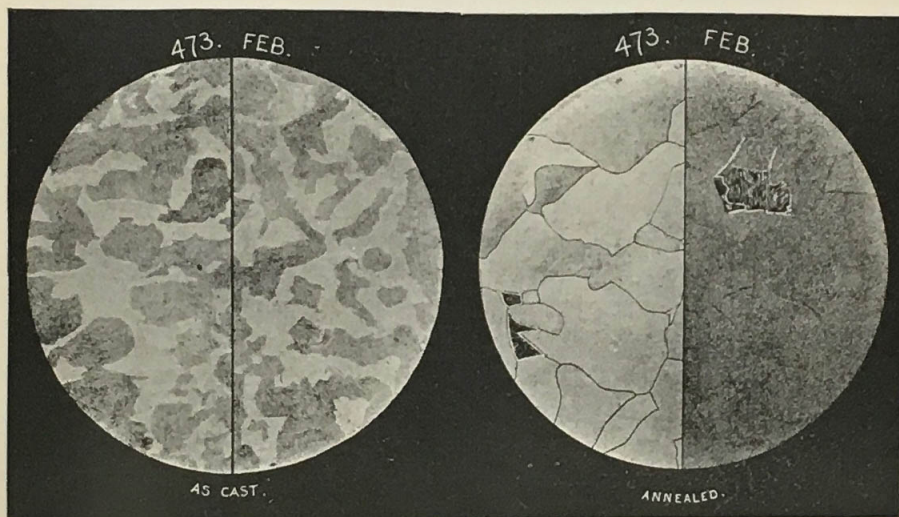


FIG. 2.—Bending Curve of Annealed Iron-Carbon Castings

because much labor has been expended on this branch of the investigation.

#### CHEMICAL, MECHANICAL, AND MICROGRAPHIC CORRELATION

The two castings FeB and 473, both of nearly pure iron, both annealed under like conditions, present specimens of those almost disheartening discrepancies which the practical steel metallurgist has from time to time to face. FeB probably constitutes a record for iron and carbon crucible steel castings.



Magnified 200 diameters.

Magnified 460 diameters.

It is, after the annealing operation, to all intents and purposes equal to forged dead mild steel. On the other hand, 473 not only gave vastly inferior mechanical results, but was not mechanically amenable to the influence of annealing, although its structure was completely changed during the operation. In view of these facts, the micrographic examination was made as thorough as possible. In the steels as cast there existed little or no difference between their curious structures. (See Micrographic key.) After annealing, the intensely crystalline structure of 473 came up with very slight etching, whilst *ceteris paribus*



# KEY TO THE MICROGRAPHIC ANALYSIS OF CASTINGS IN TABLE I

Mark	Carbon	Condition	Micro-structure
FeB	.07	As cast	Pearlite and cementite practically absent. Irregular particles of pale ferrite mixed with about equal sectional areas of dark etching ferrite, looking almost like manganiferous pearlite. These darker areas evidently contain a minute quantity of finely divided carbide. Very drastic etching was necessary to bring up this structure, which looks almost like that of a 0.45 per cent. normal carbon steel. See Micrograph 473 - FeB.
FeB	.07	Annealed	as cast as cast On light etching isolated particles of laminated pearlite surrounded by and with offshoots of cementine showed themselves. The ferrite did not etch brightly, and the crystalline junctions were practically undeveloped. See Micrograph 473 - FeB. (Magnified 200 dias.) On deep etching the close crystalline joints became visible, showing here and there isolated bits of dark pearlite and streaks of cementite between the junctions. The ferrite crystals are very large, and sometimes exhibit within them parallel lines. See Micrograph 473 - FeB. annealed annealed (Magnified 460 dias.)
473	.06	As cast	Same as FeB as cast. See Micrograph.
473	.06	Annealed	Light etching brought up well-marked ferrite crystals, some pale, some gray-brown. A few isolated patches of laminated pearlite surrounded by cementite were also visible. Crystals rather small. See Micrograph. (Magnified 200 dias.) On deep etching the crystalline joints were very broad, presenting the appearance called by Sorby "loose." See Micrograph (magnified 460 dias.)
521	.18	As cast	Ground mass of small ill-defined ferrite crystals, with elongated or rounded patches of granular pearlite. Micrograph 521.
521	.16	Annealed	Ferrite crystals large and well defined. Pearlite in large isolated masses partly surrounded by cementite. Micrograph 521.
458	.37	As cast	Irregular trellis-work pattern of ferrite and pearlite.
458	.37	Annealed	Pearlite areas somewhat small and badly defined owing to segregation of the carbide striæ into massive cementite. Ferrite crystals not very large.
518	.37	As cast	Same as 458 as cast.
518	.37	Annealed	Large ferrite crystals and large well-marked pearlite areas.
CC	.42	As cast	The micro-section from this piece was lost, but from memory its structure was much as 458 as cast.

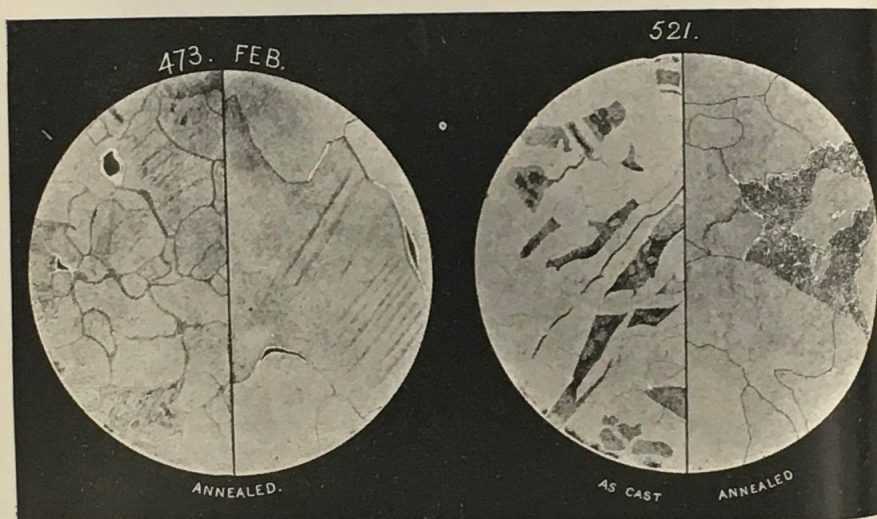
## (Continuation.)

Mark	Carbon	Condition	Micro-structure
CC	.40	Annealed	Pearlite evenly distributed in somewhat small pieces enveloped to some extent in walls of cementite.
541	.44	As cast	Much as 458 as cast.
541	.35	Annealed	Much as 518 annealed.
CC2	.48	As cast	Irregular trellis-like pattern of mixed ferrite and granular pearlite. Long lines of ferrite broken in two longitudinally by dark-brown lines of sulpho-silicide of iron (?). See Micrograph.
CC2	.50	Annealed	Well-marked and moderately large iron crystals, with large segregated patches of laminated pearlite partly enveloped in cementite. See Micrograph. In another section from this casting the lamination of the pearlite areas was not so well marked.
YB	.50	As cast	Much as 458, but pearlite area larger.
YB	.54	Annealed	Same as 518 annealed, but pearlite areas larger and ferrite areas smaller in extent.
517	.56	As cast	Ordinary trellis pattern with some brown lines of sulpho-silicide of iron (?) in the ferrite. See Micrograph.
517	.50	Annealed	Large patches of ferrite and pearlite, the latter largely laminated, but often the pearlite laminae had segregated into patches of massive cementite. See Micrograph.
556	.60	As cast	Trellis-work pattern of ferrite and granular pearlite, the latter being the greater in area.
556	.60	Annealed	Ferrite segregated from the pearlite in patches. $Fe_3C$ in pearlite showing a tendency to segregate into patches rather than laminae.
601	.70	As cast	Cells of granular pearlite surrounded by walls of ferrite. Patches of sulphide of iron concentrated into the ferrite walls were very distinct, evolving $H_2S$ on etching.
601	.72	Annealed	Pearlite as such practically absent. Carbon distributed through the ferrite in rounded globules of $Fe_3C$ .
459	.86	As cast	Ill-defined crystals of granular pearlite with small patches of ferrite. See Micrograph.
459	.80	Annealed	Laminated pearlite with some patches of white ferrite. See Micrograph.
524	.97	As cast	Granular or slightly laminated pearlite.
524	.83	Annealed	Granular or slightly laminated pearlite.
460	1.29	As cast	Cells of granular pearlite enveloped in walls of cementite. Streaks of the latter also within the cells. See Micrograph.
460	1.10	Annealed	Cells of laminated pearlite with envelopes of cementite; a few patches of sulphide of manganese visible. See Micrograph.
522	1.95	As cast	Ground mass of granular pearlite cells with envelopes, streaks, and patches of cementite. See Micrograph.
522	1.10	Annealed	Cells of ferrite dotted with little patches of cementite and surrounded by thick walls of cementite. See Micrograph.
573	1.76	As cast	Much like 522 as cast.
573	1.38	Annealed	Much like 522 annealed, except that nodules and thin rods of graphite are here and there visible.



no structure developed in FeB. On deeply etching the two under exactly the same conditions, FeB presented very large ferrite crystals with close joints, whilst 473 showed small ferrite crystals with loose junctions—that is to say, the etching acid developed broad spaces between them.

The present case is only one of many in the author's experience in which very large crystals have been associated with extreme ductility. This would seem to suggest that the condition of the joints rather than the size of the crystals is the



Magnified 200 diameters.

Magnified 315 diameters.

important mechanical factor. But, however this may be, we are dealing with an effect rather than a cause. What is the cause which produces such differing crystalline and mechanical properties? Chemical analysis being practically the same and the annealing conditions identical, there remains only one other condition not under control, and that is the initial temperature of the casting. It would almost seem that this may determine a crystalline habit which survives even the drastic operation of annealing applied to steel castings. Whether this idea is well or ill founded can only be proved when scientific pyrometry can

under practical conditions measure the temperature of molten steel, a feat which up to the present it has entirely failed to perform.

Passing to the next casting, 521, it will be noticed that increasing the carbon from .06 to .18 does not alter the elastic limit or maximum stress, and the ductility lies between the results registered for the good and bad dead mild castings, respectively FeB and 473. It will have been remarked that all the castings hitherto considered have, both as cast and after annealing, bent double cold without flaw.

A reference to the micrographs of 521 as cast and after annealing will show that, as in the case of FeB, the annealed sample presents much larger crystals of ferrite and masses of pearlite than are present in the steel as cast. Nevertheless the ductility of the annealed is distinctly greater than that of the unannealed metal.

The very mild castings hitherto considered are, however, undesirable for general constructive purposes, and suitable only for dynamo work.

The next casting of the series, namely, 458, contains 0.37 per cent of carbon, and the influence of this element now begins to make itself decisively felt by raising the maximum stress about 2 tons per square inch and much lowering the ductility, especially in the unannealed bars.

As the carbon in this casting is about the average amount employed for general work, three other castings, namely, 518, CC and 541 of similar carbon, were selected so as to make a series of four which should embody the variations met with in general practice with castings of almost identical chemical composition. Unfortunately, upon these mechanical variations the micro-structures throw little or no light. The mechanical discrepancies in nearly pure iron and carbon castings containing about 0.4 per cent of the latter element may be thus summarized:—

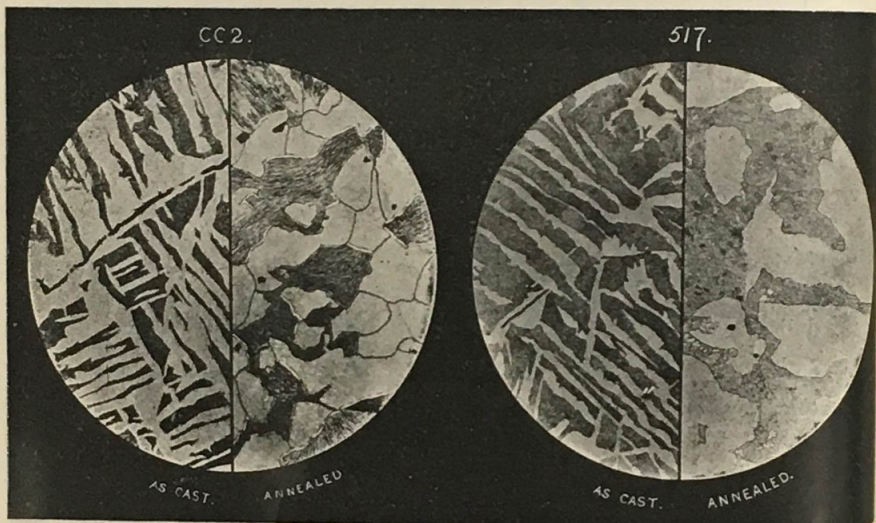
In the metal *as cast*, the elastic limit varies from 13.3 to 17.2 tons per square inch, and the maximum stress from 21.8 to 24.6 tons. The elongation per cent on 2 inches varies from 5 to 8.4 and the reduction of area per cent from 5.9 to 12.3. The bending angles range from 32° to 90°.

In the *annealed castings*, the elastic limit varies from 9 to 12.2 tons per square inch, and the maximum stress from 20.8 to 24



tons. The ductility, as measured by elongation per cent. on 2 inches, varies from 12.5 to 24.5, and the reduction of area from 16 to 29 per cent. The annealed bars all bent double without flaw.

Passing now to about  $\frac{1}{2}$  per cent of carbon, the casting CC2 exhibited very puzzling properties. The bars as cast, fractured so suddenly that no difference was observed between the elastic limit and maximum stress, although the slight elongation of 3 per cent proves that some difference must have existed.



Magnified 315 diameters.

Magnified 245 diameters.

The bending angle was trifling, being only  $12^\circ$ . On annealing, the elastic limit fell from 18 to 15 tons per square inch, whilst the maximum stress rose from  $18\frac{1}{2}$  to  $26\frac{3}{4}$  tons. The elongation gave for 0.5 per cent of carbon the high figure of 20.5 per cent, whilst the reduction of area was only 16 per cent. and the bending angle  $86^\circ$ . The result last named is quite abnormal. As a rule, a casting elongating 20 per cent will bend double without flaw. A study of the micro-sections of CC2 will reveal vital principles connected with the mechanical properties of small unannealed castings low in manganese and high in silicon.

The brittleness of the unannealed bars seems due to two main causes. First, imperfect adhesion between the long, sharp junction lines of the constituents. Although the latter exhibit sectionally a trellis-like form, their solid geometry really consists of dark etching elongated cells of granular pearlite, surrounded by pale, thick walls of ferrite. But the second cause, namely, the brown lines running almost exclusively through the ferrite, and enclosing large groups of the two constituents, is distinctly the more potent factor producing brittleness. These lines of extreme weakness will be dealt with more fully under Group B., namely, the iron, carbon, silicon series; but it may be well here to make a few preliminary remarks on their nature.

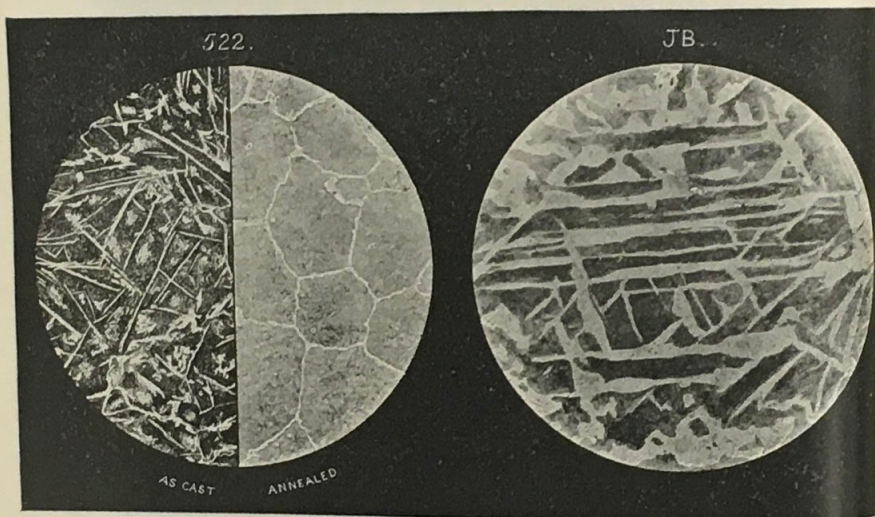
Several years ago the author showed to his friend, Mr. J. E. Stead, these curious enveloping films, and at Middlesbrough Mr. Stead independently confirmed their existence in small castings. Both Mr. Stead and the author are agreed as to their fatal mechanical effects. Mr. Stead, however, is inclined to consider them of a scoriaceous nature, that is, a readily fusible dissolved slag segregating during cooling. The author has not been able to confirm this view, because before etching they are invisible, and hence apparently of a metallic nature. The author regards them as a fusible, metallic sulpho-silicide of iron, segregating in a minutely granular form. Indeed, Mr. Stead has, by differential sieving, obtained from castings exhibiting this phenomenon a residue high in silicon and sulphur. These envelopes are dangerous only in small medium carbon castings; their more detailed description may be advantageously left until Part II. of this paper is reached, because by that time additional observations by other workers may be available to assist in deciding as to their actual nature.

In the meantime it may be remarked that the lines of these sectional meshes are often defined on etching by an evolution of  $H_2S$  gas, which covers the iron adjacent to the lines with iridescent sulphide films. On annealing these meshes are usually destroyed, running up into little isolated patches of pure sulphide of iron. (See Micrograph CC2.) The removal of these films, together with the destruction of the sharp, interconstitutional cleavage lines, largely accounts for the increase not only in ductility, but often also in tenacity observed after anneal-



ing steel castings, particularly those in which carbon and silicon constitute the bulk of the foreign elements present.

The author will in Part II. present the curious paradox that a small percentage of sulphur is actually more favorable to the production of the dangerous films just described than a relatively large amount of sulphur. With reference to the action of annealing on the carbide of 0.4 per cent carbon steels, the following explanation, which excludes ultra-scientific theories, is practically and substantially accurate. On heating the casting



Magnified 460 diameters.

Magnified 460 diameters.

in the annealing furnace to a temperature of about  $700^{\circ}\text{C}.$ , the compound pearlite areas, which consist of 87 per cent. of iron intimately mixed with 13 per cent. of normal carbide of iron,  $\text{Fe}_3\text{C}$ , pass at Osmond's point,  $\text{Ac}_1$ , into the simple constituent martensite, a highly attenuated but definite alloy corresponding to the formula  $\text{Fe}_{24}\text{C}$ . Then, as the temperature further rises between  $700^{\circ}$  and  $800^{\circ}\text{C}.$ , the points  $\text{Ac}_2$  and  $\text{Ac}_3$  are passed, and the martensite and ferrite areas gradually interpenetrate until molecular equilibrium is established and the mass is homogeneous. On cooling, the constituents ferrite and marten-

site commence to segregate at about  $750^{\circ}$  C. and at  $700^{\circ}$  are distinct, the martensite having gathered into large irregular masses, and the ferrite into allotrimorphic crystals. At about  $650^{\circ}$  C. (Ar 1) the martensite,  $\text{Fe}_2\text{C}$ , decomposes into pearlite ( $21 \text{ Fe} + \text{Fe}_2\text{C}$ ). Passing from  $650^{\circ}$  to  $550^{\circ}$  C., the  $\text{Fe}_2\text{C}$  segregates first into laminæ, then partially into imperfect envelopes surrounding the pearlite areas, and finally, if the cooling be very slow, into isolated patches; so that pearlite proper has disappeared, and the areas it formerly constituted become really ferrite containing isolated globules of cementite. Whichever stage of the annealed structure ultimately remains, the mechanical quality of the castings is greatly improved, but in varying degrees. This subject will be better finally dealt with after the data on the heat treatment of unannealed and annealed castings have been presented.

Proceeding with the consideration of the results embodied in the table, the casting YB is reached, in which the carbon is a little over 0.5 per cent. This steel, when compared with CC2, presents some remarkable mechanical discrepancies in the annealed metal. The elastic limit is only 10.5 tons per square inch as against 15.2 tons in CC2. Although the elongation per cent of YB is 4.5 per cent lower than CC2, nevertheless YB has bent through  $180^{\circ}$  against the  $86^{\circ}$  of CC2. The micrographic analysis does not seem to reveal any differences in structure capable of accounting for these curious variations. Also in the case of 517 it will be noted that an elongation of only 10 per cent is accompanied by a bending angle of  $135^{\circ}$ .

Passing next to 556, which contains 0.6 per cent of carbon, it is evident that a critical mechanical point has been passed, accompanied by a decisive falling off in ductility and in this particular casting the elastic limit and maximum stress are also very poor.

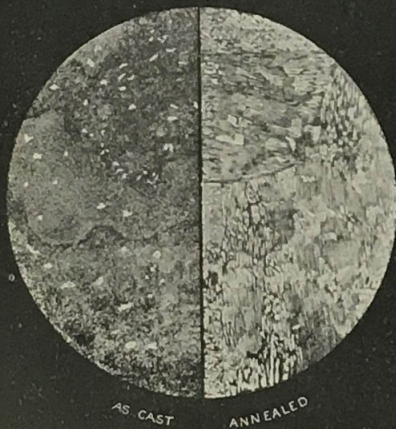
The carbon being well over the semi-saturation point (0.45 per cent), the pearlite is now the predominating constituent, and in the annealed bars of 556 the pearlite had to a considerable extent decomposed into globules of cementite. It is therefore possible that, owing to their different coefficient of contraction, these segregated cementite patches have partially detached themselves from the ferrite, thus forming innumerable flaws, which account for the unsatisfactory mechanical tests. But such a view



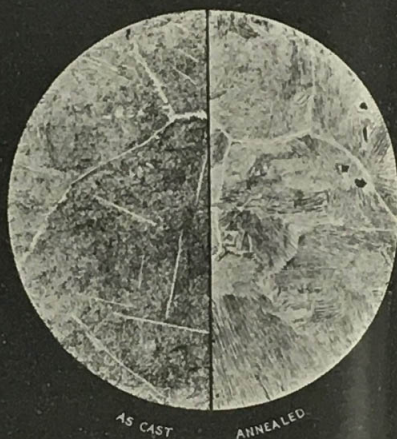
is not easily reconciled with the properties of the next casting, 601, which contains 0.7 per cent of carbon, and in the annealed sample of which the globular segregation of the pearlite laminae into cementite is even more marked than in 556. It is true that 601 has little ductility, but its maximum stress is over 30 tons, or nearly twice that of 556.

From 601, carbon 0.7 per cent, to 460 carbon, about 1.1 per cent, the maximum stress fluctuates from 24 to 30 tons, the latter evidently being the usual stress, the former the exception.

459.



460.



Magnified 65 diameters.

Magnified 65 diameters.

From 556 to the end of the series, ductility has practically vanished, if we exclude the slight recrudescence shown in the bending angle of 524, which curiously registered  $50^{\circ}$ .

522 and 573 show that very high carbon castings, both as cast and after annealing, can rank only with good gray iron castings.

The micrographs of 522 as cast and after annealing are interesting. It will be seen that during annealing a considerable amount of cementite was oxidized. In the annealed bar the residual cementite formed large cell walls, originally containing pearlite, but during the slow cooling the pearlite laminae segre-

gated into short thick plates, which no doubt, with still slower cooling, would have ultimately become isolated globules. In this case there is no doubt that, owing to their different contraction coefficients, the cementite walls partially pulled away from the cells they envelop. In fact, this section is typical of a No. 5 cemented bar.

The remarkable mechanical variations so frequently referred to make it impossible to plot curves in which carbon is coordinated with the maximum stress or elongation, but taking the annealed bending tests and excluding the obviously abnormal cases of CC2 and 524, a curve is obtained which represents with approximate accuracy the influence of carbon on annealed iron castings. The results are plotted in Fig. 2, in which the coördinates are carbon per cent and bending angles in degrees.

It will be noted that after the carbon reaches about 0.55 per cent there is a sudden drop in the ductility, the latter, so far as practical test purposes are concerned, having virtually disappeared.

The compression tests call for no particular comment beyond remarking that the capability for compression falls with the carbon in the steel as cast from 63 per cent in the nearly pure iron to 17.3 per cent with 1.8 per cent of carbon. After annealing, in the great majority of cases, the percentage of compression registered is only slightly increased, showing that the causes producing remarkable weakness in tension have relatively little effect when the material is in compression.

## HEAT TREATMENT

Heat treatment, generally somewhat vaguely called annealing, is usually only of academic interest, but in the present paper it calls for consideration in view of the unfortunate fact that experimentalists are apt to generalize from laboratory results obtained with small plain bars, and put forward their data as available for guidance in works practice. In the majority of instances such a view is erroneous and misleading. It has been more than once urged that the annealing process used by the author is unnecessarily drastic, and if in practice only little bars such as are usually employed in laboratory practice were concerned, this criticism would be sound. But such is not the case.



In large castings there often exist juxtaposed light and heavy heavy masses. The former reach the maximum annealing temperature, say  $950^{\circ}$  C. or a light red heat, long before the heavy parts of the casting are anywhere near that temperature throughout their mass, and hence before such heavy parts are in a state of thermal and molecular equilibrium. It is therefore necessary to gradually soak such castings for prolonged periods, occupying not hours but days, in order to bring both light and heavy parts to a common temperature. It is also equally necessary to allow very gradual cooling, so as to avoid highly dangerous contraction stresses, which would inevitably be set up if such castings were allowed to cool in air.

Although the cooling condition just named has in the writer's experience given the best mechanical and structural results, it is, of course, quite inapplicable to large and complex castings.

Through the courtesy of Mr. Robinson, managing director, and Mr. Jobson, chief chemist, at Messrs. William Jessop & Sons, Limited, the author is able to bring before the attention of the Institute a curious incident observed by Mr. Jobson. During the annealing of a huge marine casting, the latter, as usual, had cast upon it in various parts several test bars. All but one of these gave excellent mechanical tests. Pieces of a good and a bad test bar were sent to the author for micrographic examination, and his results exactly confirm those of Mr. Jobson. The annealed structure of the good steel (carbon 0.3 and manganese 0.6 per cent) consisted of a ground mass of ferrite, in which the areas formerly pearlite had passed into small, ill-defined particles of cementite. But the bad test bar showed the remarkable trellis-like section characteristic of brittle because unannealed castings. This structure is shown in the micrograph marked JB.

On investigation, Mr. Jobson found that this particular test bar, belonging to the bottom of the large casting, had been inadvertently buried in the sand on the bottom of the furnace; and hence its temperature had not risen above a low red heat, quite below the critical points. Therefore no diffusion of the constituents pearlite and ferrite, and consequently no recrystallization, had taken place. Hence the structure remained practically as cast and the steel relatively brittle.

The following micrographic and mechanical data give typical results obtained by varying the thermal treatment of small cast-

ings. Four  $1\frac{3}{8}$ -inch bars were cast as usual from nearly pure iron containing 0.36 per cent of carbon.

### *Casting No. 660, as Cast*

The micro-structure was of the usual trellis-like sectional pattern, but for a low silicon casting the ferrite cell walls were permeated to an unusual degree with sulpho-silicide films.

The tensile test gave the following figures:—

Elastic limit, tons per square inch . . .	13.34
Maximum stress, tons per square inch . . .	13.34
Elongation (on 2 inches), per cent . . .	1.00
Reduction of area, per cent . . .	2.20

The material was thus little better than good gray iron.

### *No. 660. Works Annealed*

The micro-structure, as usual, showed a ground mass of ferrite crystals free from sulpho-silicide films, with relatively large patches of decomposed pearlite, i. e., pearlite in which the laminae of  $\text{Fe}_3\text{C}$  had to a great extent segregated into little pieces of cementite.

The tensile test made on the works annealed bar gave the following results:—

Elastic limit, tons per square inch . . .	9.34
Maximum stress, tons per square inch . . .	22.24
Elongation, per cent . . .	14.50
Reduction of area, per cent . . .	15.00

The above is a poor result.

### *No. 660. As Cast and then Heat-Treated*

The bar as cast was slowly heated during about an hour up to  $850^\circ\text{C}$ . It was maintained for an hour at that temperature and then allowed to cool in air.

The micro-structure showed a ground mass of ferrite dotted with small particles of dark, granular ferrite. The sulpho-silicide films had disappeared, and the structure was much finer than that of the works annealed bar.



The tensile test was on the whole superior to that of the annealed bar, registering the following figures:—

Elastic limit, tons per square inch . . . .	14.34
Maximum stress, tons per square inch . . . .	26.76
Elongation, per cent . . . . .	13.50
Reduction of area, per cent . . . . .	18.60

#### No. 660. Works Annealed and Heat-Treated

Another works annealed bar was treated in the manner described for the bar as cast.

The micro-structure was very similar to that last described, but distinctly smaller in pattern.

The tensile test was not altogether satisfactory, giving the following results:—

Elastic limit, tons per square inch . . . .	15.67
Maximum stress, tons per square inch . . . .	23.77
Elongation, per cent . . . . .	11.00
Reduction of area, per cent . . . . .	9.00

From the foregoing group of results it would seem that the ideal conditions for treating a brittle 0.4 per cent carbon steel as cast are to heat it for an hour or so at a temperature about 50° above the upper critical point, and then cool in air. Unfortunately, in steel metallurgy the ideal and the practicable, are seldom synonymous terms.

#### PRACTICAL SUMMARY

The lessons taught by the data set forth in the preliminary experiments detailed in this paper show that pure iron and carbon steel is not a suitable material for fulfilling the modern specifications drafted by engineers for steel castings. With iron and carbon castings the ductility demanded can be ensured with ease, but with such ductility it is impossible to correlate the required tenacity. The latter property, it is true, can be obtained from iron and carbon castings, but at the expense of an almost complete loss of ductility. Therefore, as has already been remarked, excepting the nearly pure iron the series of castings described have small manufacturing interest. Nevertheless they form the

basis upon which the mechanical influence of silicon and manganese, to be hereafter dealt with, can alone be scientifically measured.

*The Comparative Properties of Castings and Forged Steels*

It has been previously pointed out that on rare occasions under certain, at present unknown, conditions of melting, steel castings may possess properties practically identical with those of forged steels of similar chemical composition. But such cases form the exceptions which prove the general rule, that the mechanical properties of annealed castings are much inferior to those of worked steels. This is the more remarkable because the same chemical composition, the same specific gravity, and the same micro-structure can be produced in a casting as in a forged steel, yet the mechanical properties of the latter will be enormously superior. It will be well to give concrete examples of these facts. The author, in a paper read before the Institution of Civil Engineers in 1895, fully described the properties of rolled iron and carbon steels. Comparative examples of those steels and the castings dealt with in the present paper present points of considerable interest, because the remarkable discrepancies exhibited are at present incapable of satisfactory explanation. The following tables embody the comparative properties of cast-and-rolled\* and cast-and-annealed steels of almost identical composition:—

Conditions of Steel	Carbon Per Cent	Elastic limit. Tons per sq. in.	Max. stress. Tons per sq. in.	Elongation Per Cent	Reduction of Area. Per Cent
Cast and rolled	0.21	17.08	25.39	42.1	67.8
Cast and annealed }	0.16	9.35	19.51	31.0	47.0

It will be seen at a glance that all along the line with reference to elastic limit, to tenacity, and to ductility the rolled steel is greatly superior to the annealed casting.

Passing to higher carbon the same relative characters are, if anything, still more decisively exhibited:—

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\* The rolled bars when cold were reheated to about 1000° C. and cooled in air.



Conditions of Steel	Carbon Per Cent	Elastic limit. Tons per sq. in.	Max. stress. Tons per sq. in.	Elongation Per Cent	Reduction of Area. Per Cent
Cast and rolled	0.38	17.95	29.94	34.5	56.3
Cast and annealed }	0.40	10.08	24.03	24.5	29.0

When the carbon rises still higher, the differences are still most remarkable:—

Conditions of Steel	Carbon Per Cent	Elastic limit. Tons per sq. in.	Max. stress. Tons per sq. in.	Elongation Per Cent	Reduction of Area. Per Cent
Cast and rolled	0.89	24.8	52.4	13.0	15.4
Cast and annealed }	0.83	18.5	29.0	4.0	1.7

Finally, in high carbon steel the rolled material still startlingly maintains its superiority:—

Conditions of Steel	Carbon Per Cent	Elastic limit. Tons per sq. in.	Max. stress. Tons per sq. in.	Elongation Per Cent	Reduction of Area. Per Cent
Cast and rolled	1.20	35.72	61.65	8.0	7.8
Cast and annealed }	1.10	12.86	12.86	0.0	0.0

The foregoing results should induce, in the minds of scientific metallurgists, a tinge of humility, because for some of them neither the scientist nor the practical man can offer any satisfactory explanation.

A noticeable practical feature on comparing the respective test bars is, that in the milder casting the fractures usually lack that fine gray granular appearance and cup-and-cone break which characterize mild rolled steels; also in castings the elongation is less confined to the vicinity of the fracture, being more evenly distributed along the bar, and hence accounting for the compar-

atively low reduction in area observed in castings when compared with similar rolled steels.

Castings frequently present incipient signs of fracture, i. e. small cracks, in places other than that at which the actual rupture takes place.

It will now be interesting to compare the effect of drastic annealing on steel as cast in small moulds and similar steel after rolling. On reference to the general table, it will be seen that casting 521, containing about 0.17 per cent of carbon, showed after annealing a fall of  $2\frac{1}{2}$  tons per square inch in the elastic limit, a slight fall in the maximum stress, and a decisive increase in the ductility as measured by elongation and reduction of area per cent.

Under similar conditions of annealing, a rolled steel containing 0.21 per cent of carbon showed a drop of no less than 8 tons per square inch in the elastic limit, a fall of about 4 tons in the maximum stress, whilst the elongation and reduction of area remained practically unchanged.\*

Passing to carbon 0.37 per cent, casting No. 518, it will be found that again annealing has reduced the elastic limit, in this case no less than 6 tons per square inch. The maximum stress, however, fell only about 1 ton, whilst the ductility has greatly improved, the elongation rising from 6 to 20 per cent.

On annealing a rolled steel containing 0.38 per cent of carbon, the general effect of annealing was similar to that observed in the 0.21 per cent steel, namely, the elastic limit fell from 18 to  $9\frac{1}{2}$  tons, the maximum stress from 30 to 25 tons, whilst the ductility was, if anything, slightly lowered.

With carbon about 0.9 per cent, casting 524, annealing somewhat lowered the limit and stress, and slightly raised the ductility. But on annealing a rolled steel containing about the same carbon, the elastic limit fell from 25 to 17 tons; the maximum stress from 52 to 36 tons, whilst coincidentally with this great fall in tenacity, the ductility also underwent a remarkable decrease, namely, the elongation per cent dropped from 13 to 4 and the reduction of area from 15 to 4. Hence, annealing beneficial to castings seriously injures rolled steels.

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\* See "Influence of Carbon on Iron," *Minutes of Proceedings of the Institution of Civil Engineers*, vol. cxxiii, pp. 127-162.



## THERMAL DATA

At the author's request, Mr. Andrew M'William, A.R.S.M., has kindly undertaken the recalescence investigations connected with this and the subsequent papers. His report on the iron and carbon groups is embodied in the following table. It will be more convenient to comparatively place the whole of the curves together in the final paper :—

Mark	Carbon	Heat evolved at Ar 1 on cooling	
		Rise in Pyrometric Millimetres	Equivalent Rise in Temperature of Steel
FeB	0.07	0.4	1.2° C
521	0.18	1.6	4.8
518	0.37	5.1	15.3
459	0.86	16.0	48.0
460	1.29	11.2	33.6

The heats recorded in the above table were calculated by taking the total perturbation from the fair curve (whether occurring as an actual rise, stay, or retard) in seconds, and dividing by the mean rate. The result (being the equivalent rise in millimetres) multiplied by 3.0 (the calibration factor) equals the equivalent rise in the temperature of the steel in degrees centigrade. In steels 473 and 521 and 518, Ar 1 being separate on cooling, was calculated direct from the curve. In the other two cases the total heats of recalescence were calculated, and from them the sum of the heats evolved at Ar 2 and Ar 3 in FeB was subtracted.

In conclusion, the author has to thank Mr. F. Ibbotson, B.Sc., for the patient and accurate manner in which he has reproduced the micrographs illustrating this paper.

Of the work of Mr. F. K. Knowles it is difficult to speak in moderate terms. He has for several years patiently and vigilantly carried out the details of the practical manufacture, analysis, and mechanical testing of a very large number of castings, and well merits the thanks not only of the author, but of all interested in steel founding.

## NOTES ON ALLOYS OF COPPER AND IRON \*

By J. E. STEAD

As many authoritative writers in metallurgical text-books do not agree in their remarks regarding the alloys of copper and iron, it appeared necessary to make special research in order to ascertain the truth. This has been prosecuted during the last twelve months in my laboratory at Middlesbrough.

## HISTORICAL

The records of experiments made in Dr. Percy's laboratory ("Metallurgy of Iron and Steel," 1864, p. 147) would lead any one to conclude that iron and copper could be alloyed in all proportions.

S. Rinman in 1782 made an alloy of five parts of iron and one part of copper. The alloy was hard and tough.

G. Rose found that old Roman iron finger-rings generally contain copper.

Faraday and Stodart melted steel with 2 per cent of copper without improving its quality (*Phil. Trans.*, 1822, p. 266).

Eggertz (*Wagner's Jahresbericht*, 1862, p. 9) found that wrought iron with 0.5 per cent copper showed only traces of red-shortness, whilst steel made of iron containing 0.5 per cent copper was worthless.

Longmaid took out a patent (1861, No. 1863) for an alloy of 2.5 to 10 lbs. of copper to 1 ton of iron. The patentee claimed that the iron thus made possessed unusual hardness.

In 1835 Mushet found that malleable iron united with copper in any proportion "until it equals or even exceeds the weight of copper." He found that the red color of the alloy became paler as the iron was increased above 50 per cent. The 50 per cent alloy possessed great strength. The alloy became harder as the iron increased.

Mushet not only made experiments in alloying malleable iron with copper, but also made alloys with steel and cast iron. He says steel melted with 5 per cent of its weight of copper was considerably hardened, useless for forge purposes, and incapable

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\* Iron and Steel Institute, September 1901.



of taking an edge. The ingot was crystallized like cast steel, and showed no trace of copper either on the external or fractured surfaces. Steel melted with 10 per cent of its weight of copper gave an ingot outwardly similar in appearance to the last, but with the radiated linear crystallization less distinct. It was hard and brittle, and minute points of copper visible on the fracture.

An ingot obtained by melting steel with 20 per cent of its weight of copper, when filled, appeared coppery-red on the lower and steel bright on the upper surface, and the fracture was regular in grain. Steel melted with one-third of its weight of copper (i. e. 25 per cent of the whole ) gave an ingot consisting of copper at the bottom: copper appeared in streaks and knots on the fractured surface.

White cast iron afforded nearly the same results, but there was greater tendency to separation when the copper exceeded 5 per cent. When No. 1 gray iron was melted with 5 per cent copper, specks of red copper were found upon the lower surface of the ingot and on the fractured surface; with 10 per cent copper, the copper attaches itself to the outside of the cast iron; and with 20 per cent a solid button of copper was found underneath the cast iron at the bottom of the crucible. He concluded that copper unites with iron in proportion as the latter is free from carbon.

Karsten (Percy's "Metallurgy of Iron and Steel") states that iron (it is not stated whether cast or pure iron) can only take up a certain, probably a very small percentage of copper, as copper can only combine with a small quantity of iron.

Howe ("Metallurgy of Steel," p. 368) says that Mr. Brustlein informed him that three lots of copper-steel shown at Paris by Holtzer contained from 3 to 4 per cent of copper; that with more than 1 per cent they are decidedly red-short; that they were only made as an experiment; that he believes copper-steel has no future; that the copper does not appear to be uniformly distributed through the metal; and that it appears to favor the formation of blow-holes.

H. Bauerman ("Treatise on the Metallurgy of Iron," p. 49, 5th edition) makes the statement that the two metals may be melted together in almost all proportions, but it is doubtful whether any homogeneous alloy can be prepared.

Edwin J. Ball and Arthur Wingham (*Journal of the Iron*

and Steel Institute, No. 1, 1889, p. 123) appear to have had no difficulty in alloying 4.4 per cent copper with soft iron, and 7.14 per cent in steel containing 0.71 per cent carbon, and they do not appear to have noticed any copper on the fractured surface of the alloy.

Henri Schneider (*Engineering and Mining Journal*, vol. 50, p. 40, Oct. 1890) patented a process of producing alloys of cast iron and copper by melting alternate layers of coke, cast iron, and copper, which were remarkable for their great strength, elasticity and malleability. The alloys contained between 5 per cent and 20 per cent of copper.

W. H. Greenwood ("Metallurgy of Iron," vol. i. p. 77) says the direct union of iron and copper is attained with difficulty, but an apparent homogeneous alloy can be obtained by the simultaneous reduction of the oxides of iron and copper.

J. A. Phillips and H. Bauerman ("Elements of Metallurgy," 3d edition, 1891, p. 142) make the somewhat involved statement that copper does not form any *true* alloy excepting in the presence of a third metal.

James Riley (*Journal of the Iron and Steel Institute*, No. I., 1890, p. 123), who has made experiments in alloying steel with copper, says that "if an alloy of steel and copper be made, and the resultant metal were examined under the microscope, it would be found they were not alloyed; the copper was disseminated all through the piece. If, in making that alloy, they used some aluminum, they would find a totally different result—the alloy was perfect."

F. Lynwood Garrison (*Journal Franklin Institute*, Aug. 1891) says 5 per cent copper will readily alloy with steel, but he doubts if 10 per cent will make a perfect alloy.

W. Lipin (*Journal of the Iron and Steel Institute*, 1900, No. II.) found that the addition of copper to Swedish charcoal iron increased the relative fluidity, and the fractured surfaces became more and more coarsely crystalline and brighter as the copper percentage rose. 4.9 per cent copper increased the tensile strength from 19 to 22 tons per square inch, and it did not tend to retain the carbon in the combined state. The maximum amount of copper pig iron would take up was found to be 5 per cent in both white and gray iron. He concludes that although it would not appear that there is any advantage in having copper



in foundry iron, its presence need not give rise to any apprehension.

The same author alloyed steel with varying amounts of copper up to 10 per cent, but it is not stated whether the fractured surfaces of the cold alloys showed copper specks when examined under the microscope. He found that as the carbon was increased in the steel the proportion of copper, 3 per cent, must be reduced, otherwise the material cracked during hot working.

In studying the recorded observations of the authorities who have made actual trials in alloying iron, steel, and cast iron with copper, it is not surprising that those who reviewed their work and records have had great difficulty in forming definite conclusions. All, or at least most, of the observations of the various workers are probably correct, and the reason there has been any confusion or difference in interpreting them is because no account has been taken of the influence of carbon.

#### NOTES ON METALLIC ALLOYS

Most metallurgists have their own ideas as to what a metallic alloy really is, and from time to time terms of qualification are employed, such as "true," "perfect," and "imperfect," from which we must conclude that the definition of the word is not simple. Modern research appears to show that a metallic alloy is a mixture of metallic substances, which mutually dissolve each other, and become perfectly incorporated under the influence of heat, pressure, etc., and, when in a fluid condition, do not separate into two conjugate layers, like oil and water, previous to solidification. Metallic substances may include metals, definite chemical compounds of metals with metals, of which a large number are known; definite chemical compounds of metals and non-metals, such as the carbides, phosphides, and silicides of iron and manganese. Under the definition above given, spiegeleisen, white and gray pig irons must be regarded as true alloys.

Perfect alloys may be described as those which are absolutely homogeneous when in the solid state. They consist—1st, of the definite chemical compounds of one of the constituent metals with the other; 2d, of the isomorphous homogeneous mixtures of the constituent metallic substances forming the alloys.

Intermediate in order follow the eutectics; these are not ab-

solutely homogeneous, as they are composed of separate constituents in a very fine state of division in juxtaposition.

The less perfect alloys may be described as those in which, during solidification, one or more of the constituents crystallize out in advance of the others, which eventually solidify, leaving a more or less imperfect but regular admixture in the cold metal.

When such alloys are fractured, if the constituents are of different color, they may be easily detected on the fractured surface. The purple and white constituents of the alloy of copper and antimony, containing 35 per cent copper and 65 per cent antimony, are very easily seen on the fractured surface.

If in an alloy the constituents are of the same color, the fracture appears homogeneous, but it is only a homogeneity of tint. A fractured surface of white Cleveland iron shows carbide and phosphide of iron and pearlite, but they all have approximately the same color, therefore the heterogeneous character is not apparent. If they were of different color each constituent could be detected with the naked eye.

The constituents of alloys are not the component elements present, but the separate parts, visible under the microscope.

A eutectic may be regarded as a compound constituent.

## PART I

### COPPER AND IRON ALLOYS

*Composition of material used.*—In the case of copper, a sufficient quantity of ingots of best selected copper were obtained, and these were melted and granulated by pouring into water. The analysis of the ingots was as follows:

	Per Cent
Copper . . . . .	99.7300
Oxygen . . . . .	0.0600
Lead . . . . .	0.0440
Zinc . . . . .	0.0130
Antimony . . . . .	0.0070
Gold . . . . .	0.0004
Silver . . . . .	0.0200
Arsenic . . . . .	0.0500
Bismuth . . . . .	0.0040
Nickel . . . . .	0.0470
Iron . . . . .	0.0140



In the case of iron, the coarse turnings of No. 5 British Association standard steel were employed in all the experiments where it was necessary that carbon should be practically absent. The carbon was as low, or even lower, than had been previously met with in any commercial soft steels. It gave on analysis:

	Per Cent
Iron . . . . .	by difference 99.542
Carbon . . . . .	„ 0.035
Manganese . . . . .	„ 0.317
Silicon . . . . .	„ 0.008
Sulphur . . . . .	„ 0.036
Phosphorus . . . . .	„ 0.042
Copper . . . . .	„ 0.020
	<hr/> 100.000

*Method of making the Alloys.* — Mixtures of the granulated copper and iron borings were simply melted together in clay crucibles free from carbon. The furnace employed was of the ordinary “crucible melting” type, with gas coke as fuel. It was capable of giving heat sufficient to melt pure iron. The mixtures were allowed to solidify and cool in the crucibles. When making trials to ascertain whether or not the metals separated from each other previous to solidification, the system adopted was as follows:—A series of porcelain tubes  $1\frac{1}{2}$  centimetres internal diameter and 5 centimetres in length, closed at one end, were placed side by side in a plumbago crucible. The spaces between the tubes were filled in with silver sand. The various mixtures were placed in the tubes, the lighter metal invariably being placed at the bottom. When filled, the tubes were closed with plugs of plastic ganister, and a covering of the same material was placed over tubes and the top of the crucible.

After drying carefully, the crucible with contents was heated for an hour to a heat sufficient to melt pure iron. The lid of the furnace was then removed and the fire allowed to burn itself slowly out, without disturbing the crucible, which in twelve hours was cool enough to handle. The little cylindrical ingots were then removed from the porcelain tubes and were sawn vertically through their centres, or, if too hard to saw, were ground down on an emery wheel so as to expose vertical sections of the solid metals. These, whether ground or sawn, were polished and systematically examined throughout their entire length.

The result of the examinations showed at once whether there had been any separation into two conjugate fluid layers, or if there had been any tendency to such a separation.

The system adopted of maintaining the metals in a fluid state above their melting-points and then allowing them to cool and solidify very slowly, offered the best possible conditions for separation, and if there was any tendency whatever for one metal to disentangle itself from the other it would be detected.

In order to be absolutely certain that analysis and structure were accurately correlated, the micro-sections after examination were sawn into two parts; one part was retained as a micro object and the other was analyzed.

The method of analysis used was as follows:

About 0.5 grammes of the alloy was dissolved in the least possible quantity to effect solution of nitro-hydrochloric acid, and when this was complete the solvent acids were removed by heating with strong sulphuric acid. The copper was separated by hydric sulphide and the iron determined in the filtrate by a standard bichromate solution. The cupric sulphides were dissolved in nitric acid and the copper determined volumetrically by the iodine method, a method which has been found by long experience to give quite as accurate results as the most refined electrolytic methods. When carbon was present, it was determined by burning the residue left on solution of the alloy in acid cupric potassium chloride, or by direct combustion in oxygen. Silicon and other metalloids were determined by the usual accurate methods employed in testing steels.

#### MICRO-CHEMICAL RESULTS OBTAINED

In reviewing the results of very many experiments, it was found that:

1st. — Copper and iron alloy in every proportion by direct fusion, and in none of the alloys is there any tendency for the metals to separate into two conjugate liquid layers.

2d. — That the complete series of alloys may be classed into three distinct section —

A — Alloys with traces to 2.73 per cent iron and 97.20 per cent copper.

B — Alloys with between 2.73 per cent iron and 97.20 per



cent copper and 92.0 per cent iron and about 8.0 per cent copper.

C — Alloys containing between 8.0 per cent and traces of copper.

All these mixtures may be called true alloys, being free from globules and knots of copper or iron.\*

*Class A*, containing between traces and 2.73 per cent iron, are apparently homogeneous, and contain not more than one micrographic constituent. They all have the appearance of pure copper. As the iron is increased, the alloys become slightly harder. They can be cut and sawn, and are slightly attracted by the magnet. They may be classed as perfect alloys, consisting of isomorphous crystal grains of iron and copper.

*Class B*, containing between 8.0 per cent and 97.20 per cent copper. As soon as 2.73 per cent iron is exceeded, the cold alloys are found to contain a separate constituent, consisting at first of six-rayed crystallites of a constituent rich in iron. As 10 per cent iron is approached these crystallites change their form and assume the dendritic or cruciform character of octohedral skeletons. As the iron is further increased, these crystallites also increase, and eventually mutually interfere with their independent growth by overcrowding, and assume the form of rounded crystal grains separated by envelopes of copper containing 2.73 per cent iron in solution. These envelopes become thinner and thinner as the alloy with 90 per cent iron and 10 per cent copper is approached, and at that point only partially envelop the crystal grains.

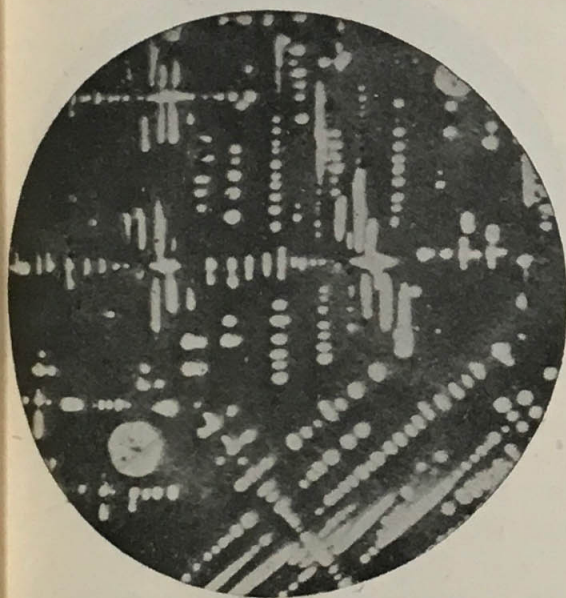
In all the alloys of this class, fracture generally follows the cupreous envelopes, and on this account there is not much difference in the color of the fractured surface of the alloys containing respectively 90 per cent and 20 per cent copper. In the alloy with 10 per cent copper, as the copper only partially envelops the grains, the fracture follows through the copper, and

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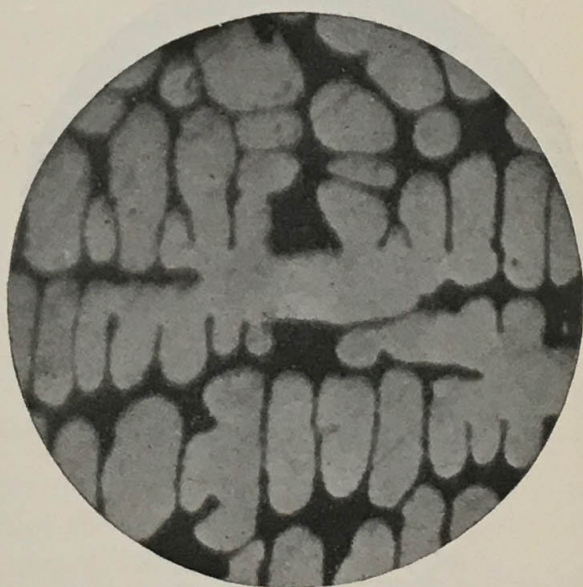
\* It was very difficult to obtain iron alloyed with a little copper without getting into the alloy small quantities of silicon or dissolved oxide of iron. When silicon was absent, some oxide of iron was present. An alloy with a little silicon and 9 per cent copper appeared to contain no free copper. When oxide was present, free copper appeared in the alloy with 8.4 per cent copper.

The determination of the exact critical point is still wanting, and the proportion 8 per cent must only be regarded as approximate.

then through the cleavages of the iron grains, leaving a macro-structure, i. e., visible without the microscope, on the fractured surface of large areas of copper and iron distributed irregularly over it, an appearance which might easily lead to the conclusion that the copper and iron were really not alloyed. There can be little doubt that observers in the past, judging only by fractures, have been deceived thereby. When the alloys are polished the appearance is quite different: the color gradually changes from that of pure copper to that of iron as the iron is increased, and



No. 1.—125 diameters. Copper, 75.5 per cent; iron, 25.2 per cent. Structure developed by polishing. White parts—Iron-copper constituent. Dark parts—Copper-iron constituent.



No. 2.—125 diameters. Copper, 53.0 per cent; iron, 46.3 per cent. Structure developed by polishing. White parts—Iron-copper constituent. Dark parts—Copper-iron constituent.

the alloy containing 10 per cent copper and 90 per cent iron, which by fracture appears to contain much copper, has on the polished surface the appearance to the eye as if copper was absent.

Practical men will readily understand the reason of the same thing appearing so different by comparing the surface of a brick wall and the appearance of the same mass of masonry after it has been broken to pieces. The surface of the wall at a little distance appears red, whereas at the same distance the fractured surface appears to be half white and half red, the fracture having

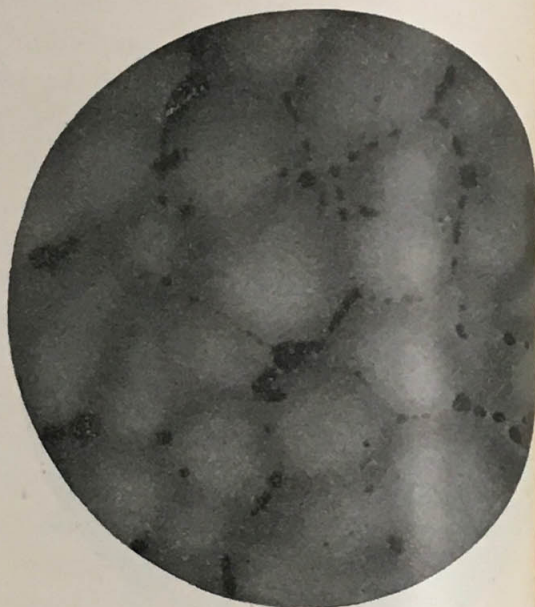
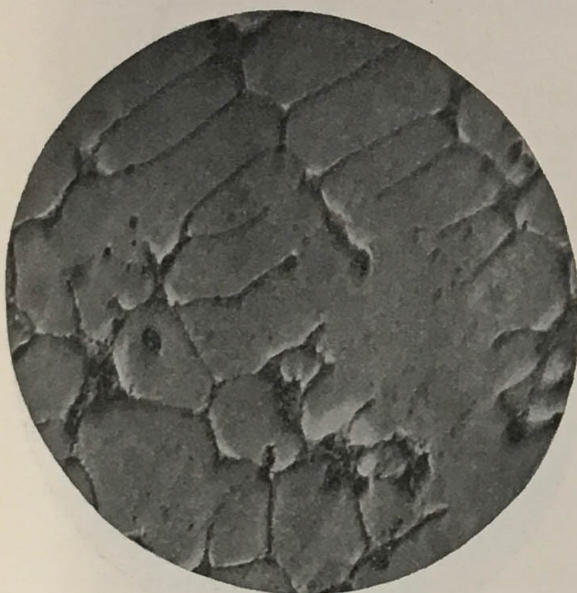


passed through and left exposed the mortar which formed a partial envelope to the bricks.

The alloys of this class increase in magnetic properties with the increase of iron.

The iron crystallites are easily rusted, and the surfaces of the polished alloys soon become tarnished and dull.

*Class C.*, containing between 8.0 per cent and traces of copper.—When iron is alloyed with about 8.0 per cent of copper, the



No. 3.—125 diameters. Copper, 20.5 per cent; iron, 79.1 per cent. Structure developed by polishing. White parts—Iron-copper constituent. Dark part—Copper-iron constituent.

No. 4.—125 diameters. Copper, 10 per cent; iron, 89.6 per cent. Structure developed by heat-tinting. White parts—Iron-copper constituent. Dark parts—Copper-iron constituent. Showing graded character of the crystal grains higher at their centres than at the exterior parts.

cold alloy when fractured shows no trace of copper on the broken faces, and the polished metal appears equally free from any copper-colored constituent when examined under the microscope, and all the alloys containing less than that proportion of copper appear equally free from it.

On heat-tinting or gently heating the polished surfaces until they assume a pale yellow tint, the crystal grains are not colored equally. Oxidation commences at the external boundaries and travels towards their centres.

It was further observed that under like heating the alloys containing the most copper are the more rapidly colored. This being so, it appeared reasonable to conclude that in the metals in this class when solidifying, the first part to fall out of solution contains the most iron, and as growth proceeds the liquid phase becomes more concentrated in copper; also, that as the crystals continue to grow, more and more copper falls out of solution with the iron until the last portion solidifies with a maximum proportion of that element. When the amount of copper exceeds about 8 per cent the iron-copper crystals, in completing their growth, throw entirely outside of them the excess of copper-iron alloy which they could not retain in solution.

The photograph Fig. 4 of the 10 per cent copper alloy illustrates the foregoing remarks very fully. The dark patches represent the copper-iron thrown out of solution, the shaded parts a gradual change in composition of the crystal grains from the external parts high in copper to the internal parts rich in iron.\*

## PART II

### THE INFLUENCE OF CARBON ON ALLOYS OF COPPER AND IRON

The results of melting copper and iron containing varying proportions of carbon fully confirm the observations of Mushet that copper unites with iron in proportion as the latter is free from carbon. On repeating the experiment of that investigator, and melting 5 per cent and 10 per cent copper with steel of 1 per cent carbon content, it was found that the former alloyed perfectly, no free copper being detected on the fractured or polished surfaces of the cold steel, and that the latter, with 10 per cent copper, showed knots of a copper-colored constituent on the broken ingots and polished sections. It was further found that steel with 1 per cent carbon would dissolve and retain in solution about 7 per cent copper, and that when this amount is exceeded, the excess is thrown out of solution at the setting point and appears as globules, or the knots of Mushet. These drops or globules are in greatest quantity near the lowest part of the ingots containing 10 per cent copper, but are not separated from

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\* These graded crystal grains are what Professor Roozeboom would describe as "mixed crystals."



the steel itself. Their form and position lead to the conclusion that they were liberated when the steel was in a semi-fluid state, and in virtue of their greater gravity traversed the mass towards the bottom and were mechanically held in suspension and prevented from escaping altogether when the steel completely solidified.

When the proportion of copper is increased above 10 per cent, the suspended globules also increase, and when 25 per cent copper is melted with 75 per cent steel, a portion of the copper separates from the steel before it solidifies, and is found at the bottom of the ingot in a separate layer. It is, however, not pure, but is associated with about 10 per cent iron, part of which is in solution and part as dendritic crystallites.

The upper steel layer contains globules of copper, which increase in quantity from the surface downwards.

All these steel alloys are too hard to cut, saw, or file.

The copper in passing into solution does not cause a separation of free cementite.

By "oxidation heat-tinting" the alloy with 7 per cent copper and 0.93 per cent carbon, the color tints indicate that the primary crystal grains contain the most copper near the external boundaries. The secondary pearlite grains are differently tinted, leading to the conclusion that different individuals may contain different amounts of copper.

On etching with tincture of iodine or dilute nitric acid, the same differences were developed.

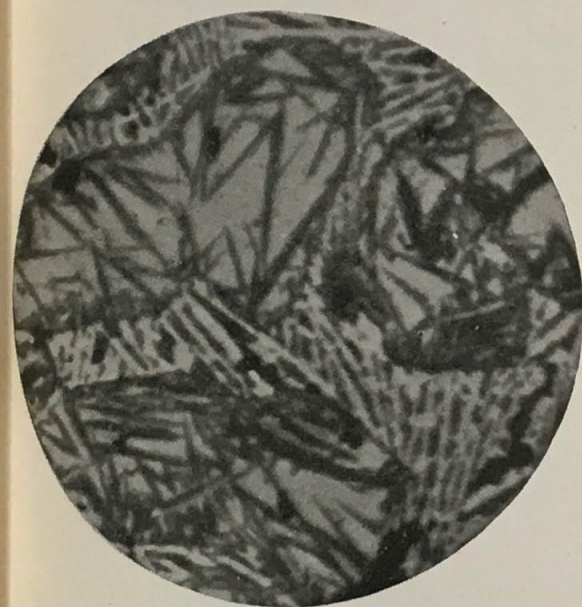
Although this alloy was very slowly cooled, the pearlite structure was very fine indeed, the lamella of cementite being very thin and close together.

When pig iron containing 3.4 per cent carbon and about 3 per cent silicon was melted with an excess of copper, two liquid layers separated, the first consisting of a layer of copper containing about 8 per cent iron at the bottom, and a layer at the top of dark gray iron, containing 7.4 per cent copper, in which globules of copper are embedded. The amount of copper retained as globules varies with the relative rapidity with which the metal is cooled, being at a maximum when rapidly cooled, and a minimum when the cooling is protracted.

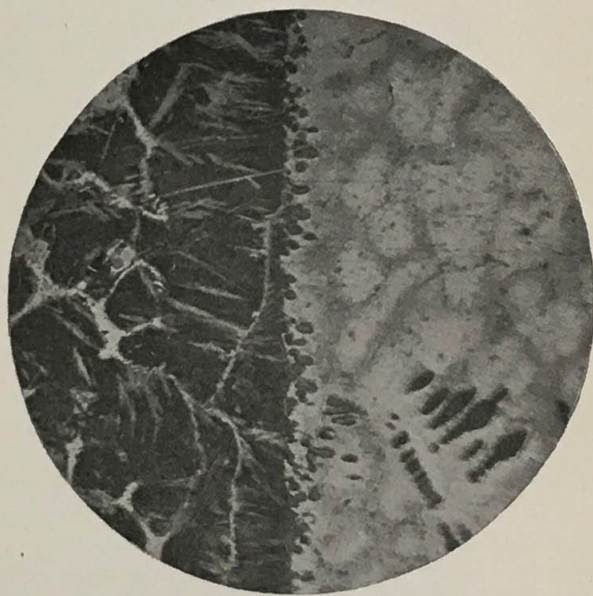
When an alloy containing equal parts of copper and iron was melted in a charcoal-lined crucible, and was allowed to cool

therein, it separated into two conjugate layers of the following composition :

	Top layer Per Cent	Bottom layer Per Cent
Iron . . . . .	87.00	9.60
Copper . . . . .	10.34	90.02
Carbon . . . . .	2.07	0.08
Silicon . . . . .	0.45	Nil
etc. . . . .	0.14	0.30
	<hr/> 100.00	<hr/> 100.00



No. 5.—125 diameters. Copper, 7.64 per cent; iron, 89.0 per cent; carbon, 2.30 per cent; silicon, 0.84 per cent. Etched by iodine. Course banded envelopes—cementite. Zigzag dark parts—martensite structure. White ground apparently austenite.



No. 6.—Junction of two conjugate layers of 50 per cent iron, 50 per cent copper alloy after melting with charcoal,  $V \times 80$ . Etched with nitric acid. The lower layer is the copper-iron alloy; the upper layer is the iron-copper-carbon alloy.

These results most clearly show that the carbon, on combining with the iron, or a portion of it, throws a large part of the copper out of solution, which then gravitates to the bottom of the crucible. They also show that the separated copper carried with it iron containing about 0.84 per cent carbon. The microscope proved that the upper layer contained free cementite in considerable quantity, also globules of copper, and portions, between the bands of cementite, of a substance in which pearlite or eutectic cementite was apparently absent.



The copper portion contained iron-like skeleton octahedra crystallites, distributed equally through the mass, but the part immediately adjoining the iron layer was free from them, which fact would tend to lead to the conclusion that in solidifying the crystallizing force of the contiguous mass of iron exhausted or attracted from the copper nearest to it the iron crystallites which, instead of falling out of solution and remaining suspended in the copper, were attracted by the iron, with which it united. (See Photo Fig. 6.)

A portion of the upper layer was melted with charcoal to whiteness for a period of one hour, and was allowed to cool slowly. On examination it was found that a small globule of copper had separated and was attached to the lower part. This was detached, and, together with the iron part was analyzed.

The following results were obtained:

	Top layer Per Cent.	Bottom globule Per Cent.
Iron . . . . .	89.00	2.73
Copper . . . . .	7.64	97.10
Combined carbon . . . . .	2.30	Nil
Graphite . . . . .	Nil	Nil
Silicon . . . . .	0.84	Nil
	<hr/> 99.79	<hr/> 99.83

There is here evidence that when copper is in solution in iron, it limits the quantity of carbon the iron will absorb, for only 2.3 per cent was taken up, under conditions which, if copper had been absent, would have approached 4 per cent, or even more.

It is interesting to note that, owing to the smallness of the globule of copper, the preponderating mass of iron actually attracted out of it the whole of the iron, excepting that part of it which formed, with the copper, a saturated solid solution.

That it was due to crystallic attraction acting through a short distance on a small mass was proved by increasing the mass of the copper, and, with that exception, conducting the trials under exactly parallel conditions. The copper separated contained nearly 10 per cent of iron, and the microscope revealed quantities of iron crystallites throughout, except at the parts adjoining the iron layer.

The small globule containing 2.73 per cent iron under the

microscope appeared quite homogeneous, and was free from iron crystallites.

The iron portion contained free cementite in net-like formation, enclosing areas, or cells, which, on polishing on parchment moistened with sulphate of ammonia solution, yielded a structure identical with the finest examples of austenite and martensite, produced by Osmond. Photo Fig. 5 illustrates this. The zigzag structure corresponds to martensite; the half-tone ground corresponds to austenite; the high white parts, cementite. On scratching this with a needle, it did not appear that there was any sensible difference in hardness in the dark and half-tone parts. They were apparently equally grooved by the hard steel.

The specimen was far from homogeneous. Near to where the copper globule had been attached, the cementite disappeared and the cells or grains consisted of almost a pure martensite structure. Whether these structures indicate either martensite or austenite has not at present been determined with certainty. More investigation is still required.

When a small portion weighing about 2 grammes was heated to and quenched from  $900^{\circ}$  C., the inter-cementite areas had a similar structure to the same areas of the slowly cooled material, with this difference that the zigzag martensite structure on the light-colored ground was much finer and more equally distributed, an observation which would lead to the conclusion, that whatever the dark substance was, it must have diffused and separated out again on rapid cooling.

If it is eventually demonstrated that the white ground mass is actually austenite, it will prove of unique interest, for hitherto that constituent has only been obtained in high carbon steels after quenching from an elevated temperature, whereas in this instance the most pronounced structures were obtained in the slowly cooled alloy.

In order to ascertain whether or not copper, when added to molten pig iron, has any influence in retarding or accelerating the separation of graphite when it is allowed to solidify in a mould, the following experiment was made: Hematite pig iron low in silicon, containing a heart of white and envelope of gray iron, specially selected as representing a condition on the borderline between gray and white iron, and therefore very susceptible to influences tending to cause the carbon to separate or to re-



main in the combined state, was melted with about 5 per cent of copper in a crucible. The metal was poured into a mould, and when cold was broken and the fracture examined. It had a fine gray appearance, and was quite free from any patches of white iron. The combined carbon was 1.20 per cent. This result appears to confirm the statement of Lipin, that copper does not tend to make gray iron into white iron, and on that account need not be feared if it is present in foundry pig iron. Judging from what we know of the effect of copper on iron and steel, it is also certain, as Lipin observes, that it increases the tenacity of pig iron when it is alloyed in it.

#### SUMMARY

In summarizing the results referred to in the foregoing remarks, the following facts may be noticed:

1. That carbon when present limits the amount of copper which can be alloyed with the iron, the maximum of 7.5 per cent being the limit in gray hematite iron containing 3 per cent silicon, and about 15 per cent in steel with 1 per cent carbon; but in most of the carbon-iron-copper alloys some of the copper exists as globules, and is only mechanically suspended in the solid metal. The amount of this mechanically suspended copper varies with the rates the metals are solidified, being highest when rapidly and lowest when very slowly cooled. The actual amount retained in solution when solid has not been fully determined. In the steel the amount does not probably exceed 7 per cent.

2. That alloys with about equal parts of copper and iron, which do not separate into two conjugate liquid layers before solidification, when remelted at a white heat on charcoal absorb carbon, and then do separate into two liquid layers, one containing about 2 per cent carbon and 10 per cent copper, the heavier about 10 per cent iron and 0.08 per cent carbon.

3. Copper in cast iron does not appear to have any influence in retaining the carbon in the combined form, or in causing a separation of graphite.

4. The micrographical study of steels containing copper leads to the conclusion that the copper retards the formation of pearlite. The same steels, differing only in copper, under like comparative rapid cooling, when cold contain more diffused carbide when copper is present than when absent. On very slow cooling, the

carbides appear to separate equally in both the cupreous and normal steels. This peculiarity explains why annealing and slow cooling have the marked softening effect referred to by Lipin and others on cupreous steels.

5. That copper in foundry iron need not be feared, as its only effect appears to be that of raising its tenacity.

6. That the somewhat conflicting statements of the authorities in our text-books appear to have been due to the fact that some of them may not have taken into consideration the effect of carbon in preventing copper alloying with iron. Pig iron will only alloy with small quantities of copper, whereas pure iron will alloy with any proportion.

There is much still to be learned and work to be done before we can say we know all there is to know on the subject of copper-iron-carbon alloys.

I have to acknowledge the valuable assistance of Mr. John Evans and of Mr. D. Robinson, who have assisted in the making of the alloys and their micro-examination.

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## ON THE RESULTS OF CHILLING COPPER-TIN ALLOYS\*

By C. T. HEYCOCK and F. H. NEVILLE

IN the Third Report of the Alloys Research Committee, published in 1895, Sir W. Roberts-Austen gives an appendix, by Dr. Stansfield, containing an extremely interesting series of cooling curves of the copper-tin alloys. These curves made it evident that for many percentage compositions there were three or even four halts in the cooling due to separate evolutions of heat, and that some of these changes must have occurred when the metal was solid. A freezing-point curve was also deduced from the cooling curves. The report contained interesting remarks on the meaning of the curves, but a satisfactory explanation at that time was not possible. In June, 1895, Professor H. Le Chatelier also published a freezing-point curve, giving the upper points only. These two curves agree in locating a sin-

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\* Proceedings of the Royal Society. Vol. 68.



gular point near the composition  $\text{Cu}_4\text{Sn}$ , but do not give any singular point nearer to the copper end of the curve.

In 1897 we also gave, in the "Philosophical Transactions," a freezing-point curve of these alloys. This curve was inferior to Dr. Stansfield, inasmuch as it gave no information concerning the changes that go on in the solid metal, but it gave a more accurate statement of the upper freezing points than had been given before. In particular it pointed out a new singular point at 15.5 atomic per cents of tin, the point marked C in the figure

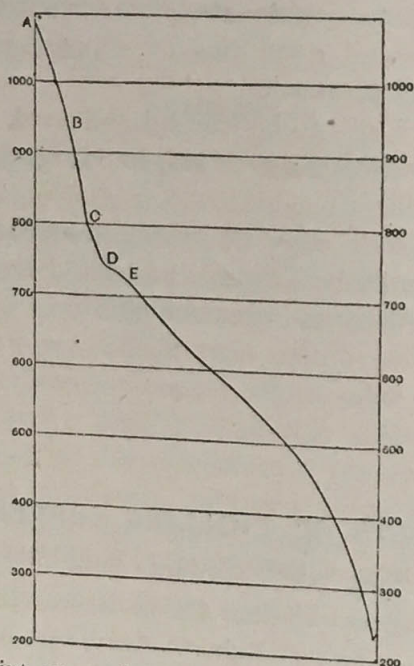


FIG. 1.—Freezing-point curve of the copper-tin alloys. Atomic percentages of tin are reckoned from 0 per cent. on left to 100 per cent. on right of diagram. (Extracted from 'Phil. Trans.' A, vol. 189, p. 63.)

(Fig. 1), and a straight branch of the curve joining C to the other singular point marked D in the figure. Both C and D are the origins of rows of second isothermal freezing points, better called transformation points. Like Dr. Stansfield, we found it impossible to offer a satisfactory explanation to the curve, but we hazarded the surmise that the steepness of the branch ABC might be due to chemical combination, and that in the region CDE solid solutions existed. Both of these surmises have since been confirmed, but at that time we felt no certainty on the subject.

In their report on alloys presented to the Congrès International de Physique in 1900, Sir W. Roberts-Austen and Dr. Stansfield give a curve embodying all the above-mentioned details and some others, in particular a most important lower curve of changes that take place in the solid alloys.\*

It may be remarked that the freezing-point curve forms a useful chart to the general character of the alloys. For example, alloys whose composition lies in the region AB of the figure are red bronzes and gun metals, tough, but not very hard, while as we approach C the alloys become paler in color and much harder. Alloys a little to the left of C are nearly white and extremely tough and strong; they are ideal bell metals. The moment we pass C the alloys begin to become brittle, and the brittleness becomes very great near D. The alloys between C and D are steel-colored; they have a glass-like hardness and take a fine polish; they are speculum metals, Lord Rosse's being the alloy at D. With more tin than that present at the point D the alloys deteriorate from a mechanical point of view, and except as anti-friction metals are not much used.

In 1900 we commenced a study of these alloys by means of the microscope. As regards the regions ABC and that to the right of E we at first did little more than confirm results which we found had been already published both by Mr. Stead and by M. Charpy; but in the region CDE we appear to have observed more detail than is contained in the published work of these observers. We were especially struck by a discrepancy, in the region CD, between the crystals on the outside of the alloys and the internal pattern. Our habit was to make the alloys in an atmosphere of coal-gas or hydrogen, and to allow them to cool in this atmosphere. If made in this way, we found that all alloys, from A almost to D, showed on the top of the ingot a regular crystallization in relief, of the rectangular comb-like character so often seen on the surface of cast metal. This was

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\* Our attention has been called to the fact that the copper-tin curve given by Roberts-Austen and Stansfield in the International Report on Physics in 1900 had already been published by them in the Fourth Report to the Alloys Research Committee in 1897. This correction does not alter the chronological sequence as stated in the text, since our paper was read before the Royal Society in June 1896.



as perfect in the white metals between C and D as in the red alloys between A and B. These crystals disappear when the point D is reached, although with much more tin other types of raised crystals are seen. These combs are of course primary crystals, standing out on account of the contraction of the solidifying mass and the consequent retirement of the mother liquid. When the ingots of alloy are cut, the surfaces polished, and the internal pattern brought out by ignition or etching, one sees, as Charpy and Stead have shown, that similar combs, rich in copper, occur in the interior of the ABC alloys, the combs being embedded in a matrix which is itself complex (see photo. 1.) These combs are numerous and large in the gun-metals of the region AB, but decrease in numbers, size and perfection as we approach C. For some distance to the left of C they are much broken and distorted, and to the right of C they do not appear at all in the body of the alloys: but they exist on the outside in the same perfection as before. Moreover if the top of one of the alloys anywhere between a point a little to the left of C and the point D be slightly ground down so as to obtain sections half through the raised crystals, and the pattern examined, it is found that the crystals are not homogeneous, as one would expect a crystal to be, but that each crystal is full of a well-marked pattern identical with that of the body of the alloy. To illustrate this peculiarity, we give a photograph of the top of the alloy containing 14 atomic per cents of tin (photo. 2). Hence it appeared that the alloys underwent remarkable changes both during and after solidification. In the alloy of photograph (2) the larger detail in the substance of the bars of raised crystal, or something not unlike it, was formed before the raised pattern, but the smaller detail, hardly seen at this magnification, is more recent than the raised pattern.

Photograph (1) shows the large primary combs existing in the interior of an alloy containing 12 atomic per cents of tin, and photograph (3) shows the utterly different pattern existing on the other side of C. It is that of an alloy containing 16.7 atomic per cents of tin. It must be remembered that on the outside the alloy still shows the combs. These alloys were slowly cooled, that is, not subjected to any sudden chill during cooling. A pattern like that of photograph (3) is given by Charpy for an alloy containing equal weights of copper and zinc. We have



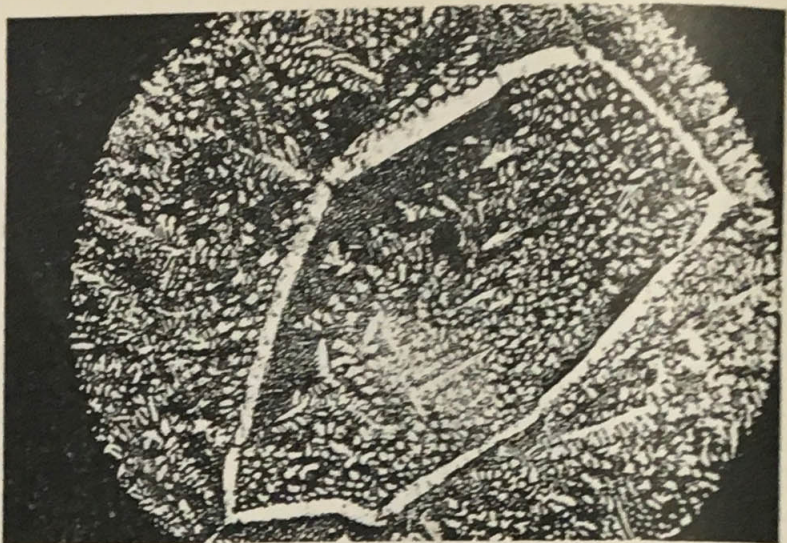


Fig. 3.—Formula  $\text{Cu}_{88.3}\text{Sn}_{11.7}$   
 Cu 72.8% Sn 27.2%  
 Magnified 300 diam. Heat-oxidized.



Fig. 1.—Formula  $\text{Cu}_{88.3}\text{Sn}_{12}$   
 Cu 79.7% Sn 20.3%  
 Magnified 50 diam. Ammonia etch.



Fig. 2.—Formula  $\text{Cu}_{86.3}\text{Sn}_{14}$   
 Cu 76.7% Sn 23.3%  
 Magnified 50 diam. Heat oxidized.



also found it in some silver-zinc alloys, and we think it always means that changes have taken place in the solid alloy.

The patterns at all points on the curve were so puzzling that we almost despaired of being able to interpret them, until after reading Professor Roozeboom's paper on the "Solidification of Mixed Crystals of Two Bodies," published in the *Zeitschrift für Physikalische Chemie* of December, 1899. The beautiful theory contained in this paper made the attempt to decipher the hieroglyphic of the copper-tin alloys more promising; but the experimental method recommended by Roozeboom, that of isolating the first crystals that form when a liquid begins to solidify, is beset with almost insuperable difficulties in the case

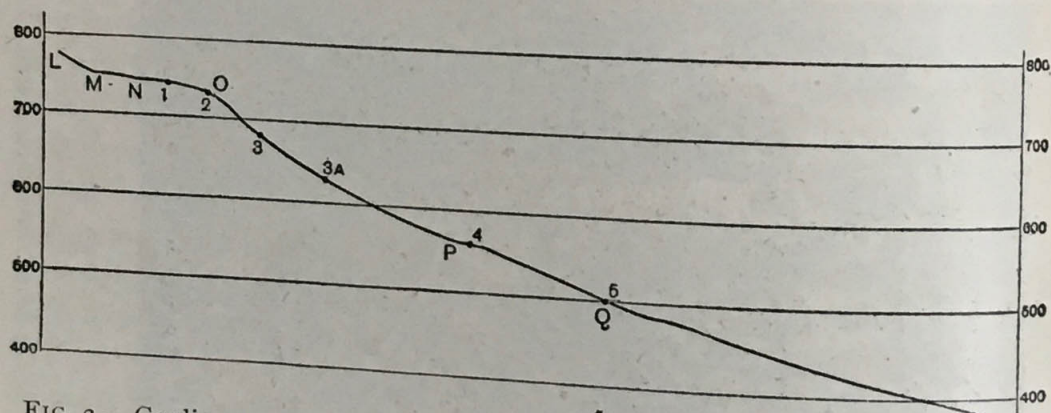


FIG. 2 — Cooling curve of the alloy  $\text{Cu}_{81}\text{Sn}_{19}$ . Percentages by weight: Cu 69.56, Sn 30.44. Time is measured horizontally. Equal vertical distances correspond to equal differences in platinum temperatures. The numbers at sides of diagram give temperatures on the centigrade scale. The numbers on the curve are the points of chilling.

of metals melting at high temperatures. Cooling curves will, it is true, give the approximate moment of complete solidification of an alloy, and enable us to plot in a rough way the "solidus" curve, as Roozeboom calls it; but the solidus curve thus obtained is not nearly so accurate as the "liquidus" or freezing-point curve. We therefore had recourse to the microscopic examination of chilled alloys, a method which has thrown so much light on the nature of steel.

The first step was to imitate Austen and Stansfield and obtain a cooling curve of an alloy by means of a recording instrument. We used a Callendar recorder in connection with a platinum pyrometer. Fig. 2 is a small scale reproduction of the cooling curve thus obtained in the case of an alloy containing 19



atomic per cents of tin. In this curve the temperature of the cooling alloy is measured vertically, and the time is measured horizontally. It will be seen that evolutions of heat occur during the period MNO and also at P and Q. Below the temperature O the alloy was a rigid mass, a solid. The temperatures marked 1, 2, 3, 3A, 4, 5 on the curve were then selected as points at which it seemed well to chill portions of the alloy. The pyrometer was therefore transferred to a bath of molten tin, heated well above the highest freezing-point of the alloy, and small amounts of from 5 to 10 grammes of the alloy, contained in little test-tubes of Jena glass, were immersed in the bath; these were in an atmosphere of coal-gas, and so did not oxidize. The bath of tin was then allowed to cool slowly and uniformly, and when the temperature fell to one of the selected points, a tube was taken out and plunged into water. The alloy was thus chilled, the slow cooling being brought to an abrupt end at any desired temperature.

The chilled alloys were afterwards ground down and polished in the usual way. After the trial of many reagents for bringing out pattern, we adopted the method of slightly heating the surface until the film of oxide formed was of a pale yellow color. Behrens some years ago recommended this method, and Mr. Stead has pointed out that it develops differences of chemical composition very well, while etching reagents complicate the picture by revealing the orientation of crystals and other details which are not always needed. With one or two doubtful exceptions, we find that in alloys richer in copper than  $\text{Cu}_3\text{Sn}$ , the parts which oxidize most rapidly, and are therefore darkest in the yellow stage, are the softer parts containing most copper. When alloys on the branch ABC are oxidized the pattern is very distinct to the eye, but it is sometimes difficult to obtain much contrast in the photographs; in such cases (for example, in the alloy of photograph 1) we etched the surface with strong ammonia, which also darkens the parts richest in copper. Alloys on the branch ABC are very sensitive to reagents such as ammonia or hydrochloric acid, and from C to D, where these have but little action, a mixture of hydrochloric acid and potassium chlorate etches rapidly. One can use these reagents to control the effect of heat oxidation in cases where the low temperature of chilling makes it possible that the heating needed to produce



the yellow color may have reversed the result of chilling; but we find that there is not much danger of such a reversal.

The upper point alloy, chilled at the commencement of solidification, was generally found to be granulated by the operation of dropping into water, but portions could always be found suitable for polishing; the other alloys had always solidified before the chilling, and therefore gave compact ingots.

After polishing, the alloys were heated until a pale yellow oxidation color was produced on the surface.

Alloy (1), chilled when much of the metal was still liquid, shows a pattern of large primary skeletons, more or less comb-like in appearance, which oxidize much more rapidly than the mother substance and which therefore contain more copper than it (photo. 4).

Alloy (2), chilled when the solidification was almost complete shows skeletons much softer in outline and not differing much in oxidation color from the ground; but these skeletons occupy a much larger area than in (1), nearly filling the field, and being only separated from each other by an imperfect network of less oxidized mother substance.

These two alloys are deeply etched in the process of polishing with rouge, the softer primaries rich in copper being eaten away. The pattern is so large that it is best examined with a power of 10 or 20 diameters.

In striking contrast to the above, alloys (3) and (3) A, chilled when the alloy has been solid some time, show no pattern even with a power of 300 or 400 diameters (photo. 5).

Alloy (4), chilled at P, the next point of heat evolution on the cooling curve, shows a pattern which is a close approximation to that of a slowly cooled alloy, and alloy (5), chilled at a still lower temperature, is an almost perfect reproduction of the slow-cooled pattern (photo 6). It will be noticed, however, that a little below the chilling point of (5) there is another stage of heat evolution, and in harmony with this we can find one point of difference between the pattern of (5) and that of the slowly cooled alloys of the region CD. Both in these and in (5) the surface is divided into large polygons bounded by bands of a smooth material, and the interior of each polygon is more or less full of a broken fern or flower-like crystallization of the same smooth body as that of the bands. The ground in which the fern leaf lies is more easily oxidized than the material of the



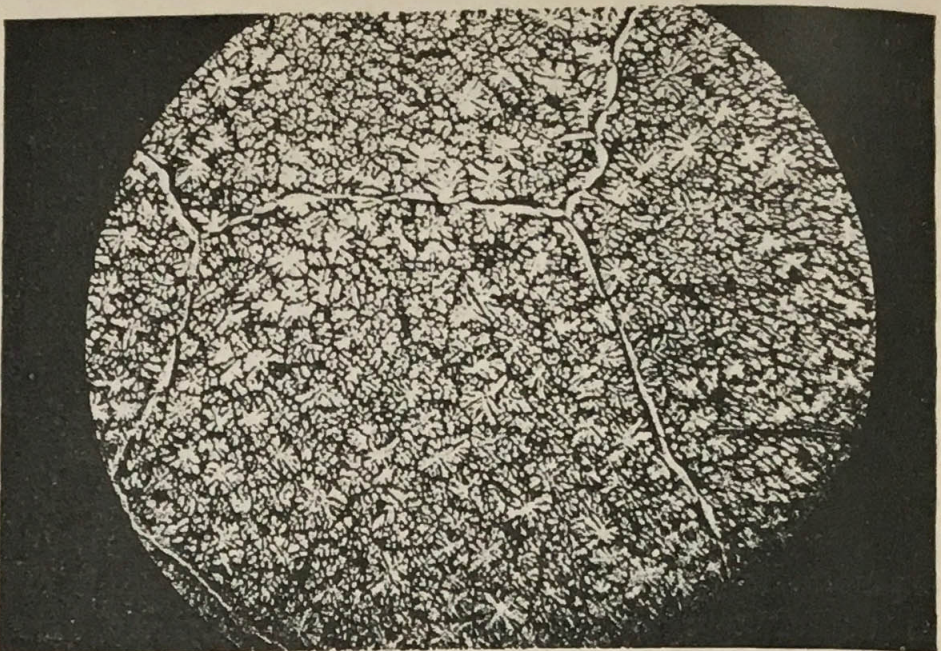


Fig. 6.—Formula same as Fig. 6.  
Chill 5  
Magnified 50 diameters. Heat-oxidized.

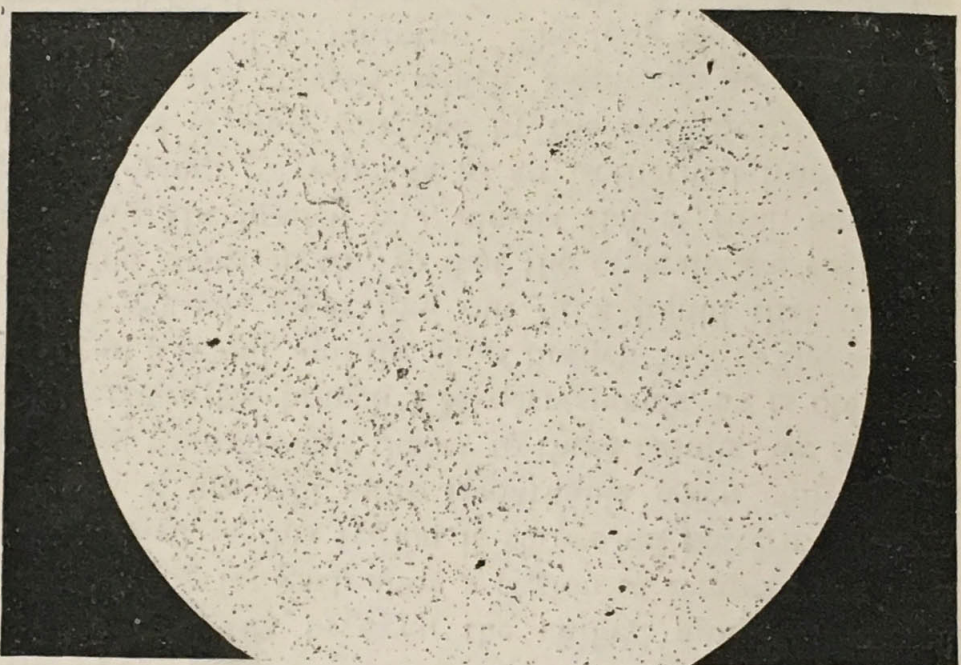


Fig. 5.—Formula same as Fig. 4.  
Chill 3  
Magnified 50 diameters. Heat-oxidized.



Fig. 4.—Formula  $\text{Cu}_{61}\text{Sn}_{39}$   
Cu 69.6% Sn 30.4% Chill 1  
Magnified 50 diameters. Heat-oxidized.



fern leaf and bands, so that the ground probably has more copper in it. In the slowly cooled alloys near C there is very little of the fern leaf, but as we approach D it increases in amount until at D it almost fills the whole area, not absolutely, however, for a network of the darker ground can still be traced here and there. A comparison of photos 3 and 6 illustrates this growth of the fern leaf with the increase in the percentage of tin. In the slow-cooled alloys the ground is granular—in fact, an immersion lens defines it as a well-marked eutetic. In (5), on the contrary, the ground appears to be uniform; probably chilling at a temperature below Q would convert it into the eutetic.

All the alloys from a little to the left of C to beyond D exhibit similar contrasts between the chilled and slow-cooled patterns, there being for each alloy a region of temperature such that if it be chilled in this region it shows no pattern. Alloys between D and E are still more remarkable when chilled.

If we apply Roozeboom's theory to these results, we see that in the cooling curve the branch LM corresponds, as is obvious, to the cooling of a liquid, and the short branch MN to the formation of mixed crystals separating out of a liquid that is continually growing richer in tin, so that the crystals are suffering transformation. The branch NO, almost flat at first, and then only slightly sloping, corresponds to an isothermal transformation of the mixed crystals followed by the solidification of the whole mass to mixed crystals, which, assuming no lag in the transformations, should be uniform. The long slope OP would then correspond to the cooling of a solid mass of uniform crystals, and therefore the alloys chilled in this region of temperature show no pattern. But at P the solid solution becomes saturated, and on cooling below this point the band and fern leaf crystallizes out. At a still lower temperature, probably Q, the mother substance of the fern leaf breaks up into a eutetic, formed in the solid. We think that P is a point on Austen and Stansfield's lower curve, and that Q is the eutetic angle of that curve. It will probably be found that the mother substance in all alloys from about B to D breaks up into a complex when the alloys cool to the temperature Q, so that if cooled slowly it is a eutetic, but if chilled above Q a homogeneous body.

It is not difficult to form a conception of how the type of pattern found below the temperature P originates. Slightly

above the temperature O the alloy consisted of crystal grains surrounded by mother liquid somewhat richer in tin. At the moment of complete solidification the grains should have adjusted themselves so as to be identical throughout, but it is improbable that so perfect an equilibrium was attained, and the solid mass at temperatures below O must have contained nuclei richer in copper than the material surrounding them. In fact, prolonged polishing brings out a vague pattern in relief, showing differences of hardness, and therefore of composition. Now the alloy that we are considering lies to the right of Austen and Stansfield's eutetic angle in their lower curve; hence when the solid solution became saturated the new crystallization commenced in the interspaces rich in tin, and more or less took their form. It is clear that the resulting structure would in section give the bands and polygons of the slow-cooled alloys. Similarly the inclusions of mother substance in the grains existing at O would be the origin of the isolated fern leaf.

Although it was hardly necessary, we thought it would be interesting to arrive at the condition of no pattern, starting from the solid alloy instead of from the liquid. We therefore took a fragment from an ingot of the same slowly cooled alloy, heated it to a faint red heat in the Bunsen flame, and dropped it into water. It showed no pattern after being polished and ignited to a pale orange. It was then heated to a temperature a little below redness, and allowed to cool for five minutes above the flame, repolished, and brought to the orange state. It then showed a very perfect slow-cooled pattern, the fern leaf being particularly good. The polygons appeared to be of the same size as in the original alloy, which had taken an hour or more to cool, but the bands were much thinner and the fern leaf smaller; the eutetic also was very scanty, while in the original ingot there were large spaces of it. Thus the same alloy, without being melted, can by heating and chilling have all pattern removed, and by reheating, followed by a not very rapid cool, the pattern can be restored. The constancy in the size of the polygons points to their having been formed at an earlier period in the history of the alloy.

We see from the above that the patterns of slowly cooled copper-tin alloys are, at all events until they have been confirmed by the examination of chilled portions, entirely misleading as to



the separations that occurred during solidification. Even the evidence for the existence of the compound  $\text{Cu}_3\text{Sn}$  will have to be revised; although in a somewhat altered form it will probably be found to be satisfactory.

We hope shortly to present to the Royal Society a more complete account of these alloys.

## WHAT IS THE ESSENCE OF CRYSTALHOOD? \*

By H. M. HOWE

**S**HOULD external form, or the polarized organization of matter of which that form is only the most striking manifestation, be taken as the essence of crystalhood?

On page 183 of the *Metallographist* for July, 1900, III. in his admirable discussion of the crystallography of iron Mr. Osmond makes the question whether a crystalline object is or is not a crystal depend on its external form, so that the fact that the matter of which the object is composed is polarized around certain definite axes, while essential to crystalhood, does not in itself constitute crystalhood. In addition, the external form must be geometrical, and a function of the internal arrangement.

The question is of especial interest to metallographists, because the crystalline entities with which we have to do rarely have a geometrical exterior, though they have all the other characteristics of true crystals. To us the question whether we shall call the grains which constitute our metallic masses "crystals" or merely "crystalline grains" or "fragments" is one of importance, and indeed of convenience.

Language looks rather to the de facto than the de jure. In our aim to keep language stable and conservative we may justly oppose changes introduced by the careless into the meaning of words; but when we try to undo changes which have already occurred our task is often hopeless. Then instead of being the conservers we ourselves are the changers. Indeed, unless the advantage to be gained is great, it is doubtful whether an attempt to reverse a change which is already well established is justifiable.

\* Received November 30th, 1901.

This is especially true when the change which we would reverse is a natural expansion, growing out of the development of our conceptions and of our knowledge.

In regard to crystalhood, then, we may ask two questions: "What is the usage of competent writers"? and "Is it desirable to change that usage"?

It is both true and natural that the older crystallographers, whose attention and indeed knowledge was chiefly confined to external form, should have based their definitions on it. Thus Dana:

*Crystal.* An inorganic solid body, bounded by plane surfaces symmetrically arranged, and produced by the laws of chemical affinity acting on its constituent molecules in the transition from a fluid to a solid state.

But modern writers, at least in this country, have often adopted the view that internal structure without external form sufficed to constitute crystalhood, and that external form was not essential to it. Thus the Standard Dictionary:

*Crystal.* A chemically homogeneous body which, in the absence of internal or external stress, is anisotropic and possesses the property of growing in a supersaturated solution.

A crystalline grain lacking external symmetry these writers call a crystal. If you ask what crystal it is, they reply it is an "anhedron," a faceless crystal.

It seems to me that this usage is so well established among competent writers that it should be accepted and assented to, unless grave reasons oppose it; that the burden of proof is upon those who would dislodge it, and would restrict crystalhood as of old to externally symmetrical bodies.

So much for the de facto. Now as to reasonableness.

Assume a case of an initially perfect octahedron growing within a supersaturated solution. This octahedron is an organized being. Just as the surface of the intestine of an animal has the power of selecting from the food presented to it those substances required for the nourishment and the growth of the animal; the power of absorbing them, of assimilating them, and of rejecting all other matter; and just as the animal is sustained and grows through the selective, absorptive, assimilative, and rejecting power of the surface of its intestines, precisely so the crystal is armed with surfaces which have the power of selecting from the



liquid in which it is immersed, those molecules necessary to its growth (namely molecules of matter like to itself) of absorbing those particles, of assimilating, i. e., of polarizing and arranging them in a definite manner about its own structure; and finally of rejecting all foreign molecules. These faces thus have the power of selection, absorption, assimilation (i. e., polarization and arrangement according to the structural needs of the organism) and rejection, exactly like the digestive and assimilating powers of the animal. Therefore I say that the crystal is an organized being, and that each crystal and detached crystalline grain, is a separate and definite organization.

Suppose that our octahedron now keeps on growing, maintaining itself as a perfect octahedron until it has grown to such a size that certain mechanical barriers which it meets within the liquid interfere with its further growth, and that the further assimilation of matter is now unable, because of mechanical obstruction, to preserve the strict octahedral form. The form then which had been geometrical up to the time when this mechanical interference occurred, now ceases to be geometrical. The object ceases to be a perfect octahedron; but does it cease to be a crystal? Are we to suppose that this organized being continues to be a crystal up to the moment of this outside mechanical interference, and that its further growth makes it cease to be a crystal? Is it not a more workable, a more reasonable scheme to hold that it remains a crystal, a deformed crystal if you please, but still a crystal? The Chinese woman's foot deformed by restraint remains a foot, a deformed foot if you please, but still a foot.

Let us take another case. Our assumed perfect octahedron is removed, and its angles are ground off slightly by mechanical means. If the grinding be very slight it clearly remains a crystal. If we continue the grinding and deface it more and more without breaking it up, the question is — do we make it a deformed and defaced crystal, or does it become, as Mr. Osmond's definition implies, only a crystalline fragment? Let us assume that we grind off from the face of such a crystal 10 per cent of its matter, enough to destroy its geometrical shape completely. Are the remaining 90 per cent a crystal or not? Should we not say that they are a mutilated crystal but still a crystal?

Or, if we take our crystal which had been distorted by mechanical obstruction to its growth, and now grind it down to a

geometrical form; do we thereby change what was not a crystal into a crystal?

I take a cube of fluorite; I begin cleaving it along its octahedral cleavage; I quickly destroy its symmetry; I continue cleaving until I have developed a perfect octahedron. At the beginning and end of this operation there is perfect external geometrical form, symmetrical with the internal structure. Shall we therefore say that the object began as a crystal, ceased to be a crystal when I began cleaving it, and again became a crystal when I finally reduced it to a perfect octahedron? Is it not a more workable and reasonable scheme to hold that it was manifestly a crystal all the time?

The case is essentially different from that of a statue, a cube or even of a man, and really goes beyond even that of a book. Statuehood and cubehood depend exclusively upon external form and absolutely upon nothing else. I knock off the nose of a statue and, while the nose becomes a fragment, the body remains a statue. I proceed, I knock off the arms and legs, and soon the statue ceases to be a statue and becomes a fragment; for, since statuehood depends exclusively upon external form, relatively slight mutilation suffices to change a statue to a fragment. In case of a man we can carry the destruction much farther before manhood ceases, because manhood depends directly on life, and only indirectly on external form as necessary to life; we can cut off both arms and both legs, and, if life continues, the man remains a man and is not a fragment merely.

In case of a book, however, mutilation can go much farther without destroying bookhood. We tear the covers off a book and it remains a book, the very same book, because bookhood is not essentially dependent upon external form. May we not say the same for the crystal — that external form is not essential to crystalhood? In crystalhood as in bookhood is not the interior far more essential than the external form? But is not the essentialness of internal organization true a fortiori of crystalhood, since the internal organization is the generator of outward symmetry, and will reproduce it if the permissive condition, immersion in a suitable solution, exists?

Is it not a more reasonable and workable scheme to regard the matter of which metals are composed as in general arranged in crystals? Each grain of ferrite in low carbon steel is a definite



organized being with definite cleavages, with its particles polarized about definite axes, and maintaining an existence independent of its neighbors; is it not more reasonable to call these grains crystals, imperfect, deformed crystals, but still crystals? Their external form is not developed because of the mechanical obstructions of their neighbors; but is it not more reasonable to admit that their internal structure, and their existence as independent entities, each developing its own structure and polarizing its own particles according to its own laws, make them crystals?

If I am right as to the reasonableness of this idea, do not considerations of convenience lead us in the same direction? If we speak of these grains as "crystals," do we not thereby help the reader to a true conception of the case, by this repeated suggestion of crystalline structure? We do not deceive him by implying external symmetry, because the illustrations before him, and indeed his first dip into the subject, show him that such symmetry does not exist. Whereas if we speak of these simply as "grains," is not the suggestion rather away from than towards the important conception of the crystalline structure and nature of these grains?

It may be said that we can meet the difficulty by calling these grains "crystalline grains"; but is there any sufficient gain to offset this encumbering of our pages? If we call these entities "crystals" we deceive nobody, but we suggest the truth. To call them "crystalline grains" is cumbrous, to call them "grains" or "fragments" is vague if not misleading.

To sum up, the basing of crystalhood on internal structure, regarding external symmetry as unessential, has such standing that it should not, as an alternative usage, be opposed unless for reasons of weight. But, far from finding such reasons, had we to choose anew between internal structure and external symmetry as the essence of crystalhood, the former would be not only the more weighty, reasonable, and workable, but also the more convenient basis.

## CRYSTALLIZATION PRODUCED IN SOLID METAL BY PRESSURE\*

By W. CAMPBELL

**I**N making experiments at the Royal College of Science on the influence of thermal and mechanical treatment on the micro-structure of metals and alloys, I noticed, in October of last year, that the effect of hammering a button of slowly-cooled tin was to produce a fine crystalline structure, the original structure of the tin being completely destroyed.

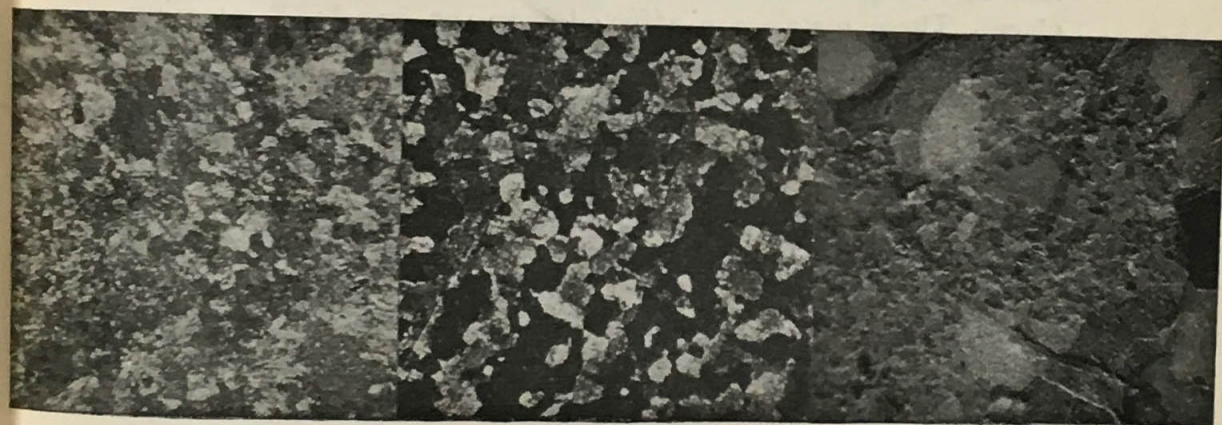


Fig. 1.

Fig. 2.

Fig. 3.

Fig. 1 is a circular section of a button of tin hammered into square form. It has been polished and etched with dilute nitric acid, is magnified 33 diameters and obliquely illuminated.

In the preparation of sections of tin, it was found that particles tend to cling to the file and, if allowed to remain, tear the surface of the metal. The effect is not immediately noticeable, but on etching the polished surface there appear besides the usual structure of the tin, lines of much smaller crystals, with irregular boundaries but possessing different orientation. If the tearing effect of the file has been extreme, these crystals may blot out the initial crystallization produced by the original cooling.

Fig. 2 represents a section of cast tin, magnified 82 diameters, vertically illuminated.

If the surface be gradually worn away by continued polish-

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\* Received August 15th, 1901.



ing, these crystals gradually disappear and the usual structure of tin appears, showing that the effect is only on the surface. A similar crystallization to that shown in Fig. 2 is set up when a section is cut with a saw. If in the process of polishing the metal thus modified is not cut away the fine crystalline structure comes out on etching.

Fig. 3 is a section of slowly-cooled tin in which this structure has not been entirely removed and the finer crystals are seen together with the larger crystallization characteristic of slowly-cooled tin.

When lead, cadmium or zinc are hammered the original structure is destroyed and a new, very fine crystallization takes its place. These metals are affected more or less by the tearing of the file and might have been used to illustrate this note. Tin, however, is most easily etched.

Thus it would appear that a metal or even an alloy will rearrange itself under the slight pressure exerted by a file.

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## ANNEALING OF STEEL CASTINGS \*

By W. M. CARR

THE purpose of this paper is confined only to the annealing of such steel as is used in the ordinary branches of the manufacture of castings. Since steel for many purposes is gradually replacing cast iron, being one of its strongest competitors, and as the output is rapidly increasing, the knowledge of its properties and the best means of handling it in the various stages of manufacture should keep step with its marvelous growth and adaptability.

The familiar quotation, "as true as steel," has on many occasions received a rude shock, leaving a doubt in the minds of its users as to the fitness of the phrase. Mention may be made of the United States Government going back to the old-fashioned wrought-iron anchors for its ironclads in view of the treacherous and mysterious failures of the most approved type, of cast

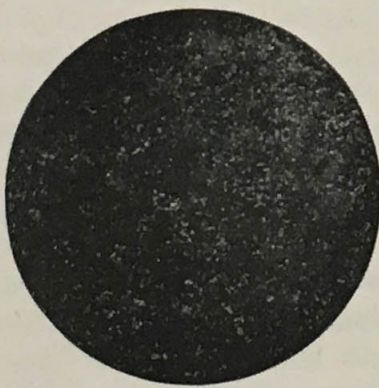
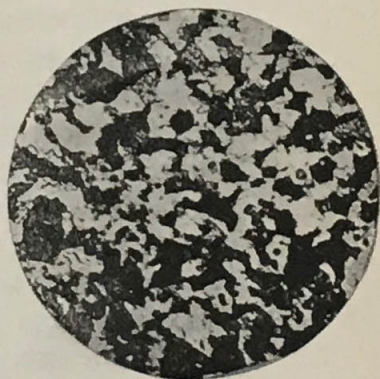
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\* *The Foundry*, October 1901. The photomicrographs illustrating this article are reproduced through the courtesy of *The Foundry*.

steel kedge anchors. Other instances could be introduced along similar lines, proving that steel may be ideal in its chemical composition, following the most rigid specifications for any given class, and yet without any warning and possibly at a most critical time may fail in service or manifest an excessive degree of brittleness. The natural question is—what causes brittleness? With abnormal chemical composition the answer might be comparatively easy, but when normal chemically the mystery becomes intensified. The question of unexplained failures has received the attention of many noble and patient investigators; each advancing elaborate hypotheses and theories with some degree of truth; but it remained for the microscope to give the



Unannealed Steel.

Properly Annealed Steel.  
Magnified 140 diameters.

Over-Annealed Steel.

true explanation leading up to the cause of brittleness in steel, and by its use, showing how such can be lessened or prevented. Herein lies the microscope's usefulness to the advanced metallurgist seeking for causes not understood in the light of the usual chemical analyses of steel castings. The writer holds to the view that brittleness in finished castings is due to a degree of crystallization as a result of imperfect heat-treatment. This being true, as proved by tests and experiments, the nearer cast steel approaches a non-crystalline or distinctly amorphous structure, the less will be the degree of brittleness, or conversely the greater the degree of toughness. It can be shown that it is possible to have two pieces of steel identical in composition, yet possessing contrasting physical properties. To the uninitiated this may seem very mysterious, but when the effect of heat-



treatment is understood the explanation is simplified by the comparative study of the structure of the pieces as revealed by the microscope. Great credit is due to Sauveur and other eminent microscopists, who by their labors have given the world knowledge of much moment to the industrial side of the metallurgy of steel.

Before advancing arguments in the support of the necessity of annealing all ordinary steel castings, it may be helpful to mention briefly the effect of heat upon the micro-structure of cast steel and its physical properties, since the relation between the two is distinct. The term "annealing" herein used is not meant that long continued heating at a white heat generally accepted as annealing, which in reality is mere soaking. Properly stated, annealing is that process of heat-treatment of cast steel by which crystallization or brittleness is removed, conferring upon the metal its best possible physical condition, the degree of heat being governed by the carbon content, and the length of time by the size of the piece. If a piece of steel be allowed to cool (not quenched) from the casting temperature its fracture will be more or less crystalline, and the micro-structure correspondingly coarse, depending upon the initial temperature and the rate of cooling. Such steel possesses fairly good tensile strength, low elongation and comparatively no resistance to impact. Upon re-heating to a suitable temperature the changes in the crystallization, if fractured, are very marked; the large ones of the original piece having disappeared, being replaced by a very fine grain. The micro-structure will be found also very fine, with the carbon compound evenly distributed throughout the mass. *Such steel will be slightly lower in tensile strength*, will be much improved in elongation and reduction of area, and all brittleness being removed, the steel can then be hammered cold, bending flat upon itself without fracture. This last-named test gives more information in the opinion of the writer as to the physical condition as a result of careful heat-treatment than can be gained by the steady pull of a testing machine, even with considerable elongation, usually regarded as an evidence of toughness, since it is known that steel can elongate and still have little resistance to shock. It is the purpose of this article to emphasize the need of careful and intelligent annealing, necessitating the dissolving of the varied opinions as

to whether steel castings should or should not be annealed when not supported by facts gained by patient trial or investigation. There is no part of the manufacture of steel castings that receives more abuse than the reheating, and it is not to be wondered that some claim that such treatment is not necessary; but when it is understood that the best possible physical properties of normal steel for a given class can only be acquired by careful heat-treatment there can be no doubt that any manufacturer of steel castings with the aim to reach the best results will be willing to give the problem serious attention.

Given a normal chemical composition, the physical properties of cast steel regarding resistance to all strains, then, are solely a function of heat-treatment; so to produce the desired and most important quality, namely, *resistance to impact*, the temperature of annealing is confined necessarily to certain limits; otherwise the attempt is defeated, since a high heating produces crystallization and its equivalent, brittleness; or in the case of too low a heating the original coarsely crystalline structure conferred by the casting temperature will not be removed. Herein lies the secret of tough steel.

The writer, as chief chemist for the Shickle, Harrison & Howard Iron Co., St. Louis, was led to take up the study of cast steel by means of the microscope. Needless to say, its value as an adjunct to a modernly equipped laboratory is inestimable. The photo-micrographs submitted are of steel made by the above firm, and are considered fairly representative of twenty-five to thirty carbon steel, and are uniformly magnified one hundred and forty diameters.

In conclusion, with the deserving attention to both the chemical composition and subsequent heat-treatment, steel castings can be made with such properties physically that will insure results in service to an extent that when not strained beyond the elastic limit of the metal they will have a life that will be practically indefinite.



## METALLOGRAPHIC NOTES

**Contributions to the Study of Alloys.** — The Société d'Encouragement pour l'Industrie Nationale, of Paris, has published a beautiful volume of 517 pages, entitled *Contributions à l'Étude des Alliages*, which includes the results of the researches undertaken by the Committee on Alloys of the Société previously published in the *Bulletin*. The list of these papers is as follows:

1. Research on Alloys of Copper and Zinc, by G. Charpy; 62 pages.
2. On Microscopic Metallography, by H. Le Chatelier; 8 pages.
3. Research on the Structure of Metals, their Genesis and their Transformations, by Sir W. Roberts-Austen and F. Osmond; 20 pages.
4. Research on the Fusibility of Metallic Alloys, by H. Gautier; 28 pages.
5. Microscopical Study of Metallic Alloys, by G. Charpy; 38 pages.
6. Magnetic Properties of Hardened Steel, by Mrs. Slodowska Curie; 44 pages.
7. Study of White Alloys called Antifriction, by G. Charpy; 40 pages.
8. Study of the Influence of Temperature upon the Physical Properties of Metallic Alloys, by G. Charpy; 36 pages.

The following important articles closely related to the work of the Committee are also published in the same volume:

9. General Method for the Micrographic Analysis of Carbon Steel, by F. Osmond; 50 pages. This memoir had been published in the *Bulletin* of the Société, but, as it appears now, it has been carefully revised, enlarged and brought up to date by its author.
10. Iron and Steel from the Point of View of the Phase-Doctrine, by H. W. Bakhuys Roozeboom; 60 pages. This is a full French translation, previously published in the *Bulletin*, of the German original.
11. On the Properties of Alloys, by H. Le Chatelier; 26 pages. This is a partly original paper dealing principally with the experimental study of the dilatation of alloys and the relation between these dilatations and the allotropic transformations corresponding to thermal critical points.
12. On the Electrical Resistance of Alloys, by H. Le Chatelier; 8 pages. This article is made up of three short notes previously published in the *Comptes Rendus*.
13. Note on the Technology of Microscopic Metallography, by H. Le Chatelier; 20 pages. This paper was originally published in *The Metallographist*.
14. Metallic Alloys, by H. Le Chatelier; 18 pages. This paper was first published in the *Revue Générale des Sciences* for June, 1895.
15. Research on Nickel Steel, by Guillaume; 44 pages. Previously published in the *Bulletin* of the Société.

16. Research on the Chemical Constitution of Steel and Cast Iron, by A. Carnot and E. Goutal; 24 pages. Published originally in the *Annales des Mines*, October, 1900.

The articles marked 5, 6, 7, 13 and 16 were translated and published in full in past numbers of *The Metallographist*, while article 11 was partially reproduced. It thus appears that nearly one half of this voluminous publication has been translated for the benefit of our readers, and we hope to be able to offer them soon a full translation of the classical work of Osmond, *Méthodes Générales*, in its revised form.

This publication of the Société d'Encouragement testifies to the success of their enterprise, and entitles them to the gratitude of the scientific and industrial world.

The typography of the book and the numerous illustrations are excellent. The price of the volume is, in Paris, 30 francs.

We reproduce below in full the preface of Mr. Linder, President of the Committee on Alloys, which explains the aim and scope of their important work:

Industrial operations cannot be properly conducted without the assistance of scientific research; it is a commonplace assertion. Empirical methods have frequently preceded theoretical rules and have even suggested theoretical studies, but these have, in turn, soon led to improvements in practical methods. Many industries can exist to-day only by calling incessantly upon science for assistance, while in the case of others empirism still remains the only guide of the manufacturer, even for the conduct of the most important operations. The preparation of alloys whose applications have always been full of interest, and which are becoming every day more numerous and more varied, belongs to the latter class.

The results of many investigations of alloys had been published, but they were not sufficient, and often presented such contradictions that it had become absolutely necessary to go over the work, completing it and presenting it more systematically.

In 1893 the Société d'Encouragement pour l'Industrie Nationale decided to lead this movement in France, first in creating a prize of 2000 francs to be awarded to the author of the most valuable investigation of the properties of alloys,\* and later in

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\* This prize was awarded in 1893 to Messrs. Osmond and Roberst, Austen.



taking the initiative in undertaking experimental researches and appointing to that effect a special committee composed of members of the council.\*

In the following pages will be found briefly outlined:

- I. The motives which led the Society to undertake this work.
- II. The experimental methods which were considered better adapted.

## I

Metallic alloys occupy an important place in the industrial world. The character of some of their physical properties (hardness, malleability, fusibility, unalterability) cause them to be preferred in many instances to pure metals. In spite of this extended use very little was known concerning their constitution and properties. To prove it, it will suffice to open at random a book on general or industrial chemistry; a few lines only will be found devoted to their subject. The result was that while numerous industries were receiving a powerful impulse from the progress of chemistry since the beginning of the century, the alloy industry, escaping this influence, remained stationary, entrenched in its old empirical methods. Each progress introduced in this industry was the result of innumerable tentative experiments, which could have been suppressed, if not entirely, at least to a very great extent, with the assistance of accurate scientific data.

Interesting scientific papers had been published on alloys, such as those of Grace-Calvert, Mallet, Matthiessen, Roberts-Austen, Lodge, Kamensky, Laurie, Riche, and others, but their conclusions, owing to their apparent contradictions, were ignored both by chemists and manufacturers. And still when they are carefully examined and compared, one is obliged to admit that while they included unmistakable errors, they have established some very important facts, such, for instance, as the existence and the chemical formula of definite compounds in certain commercial alloys (bronze and brass). They have also improved and shown the efficiency of certain experimental methods which may be applied to all similar cases, and which, if systematically applied, would surely add greatly to our knowledge of alloys.

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\* Messrs. Linder, president; Le Chatelier, secretary; Cailletet, Carnot, Carpentier, Hirsch, Sauvage, Schutzenberger, Vieille, and Violle, members.

The most salient point of these researches, considered in their ensemble, is that all the properties of alloys, and, therefore, their industrial value, depend directly upon two factors:

1. Their chemical constitution, that is, the proportion of the component metals, of the chemical compounds formed by these metals, of their mixtures and of their various allotropic conditions.

2. Their physical constitution or structure, that is, the shape and dimensions of the crystals of the elementary constituents, which, through their juxtaposition, constitute the metallic mass.

The influence exerted by the chemical constitution upon the properties of alloys is very great.

A. It has been shown long ago that the great hardness of bronze, of aluminium bronze and of brass is due to the formation, in these alloys, of very hard definite compounds, whose existence and composition have been ascertained by the investigations mentioned above. The case is very different with alloys of tin and zinc, of lead and aluminium, which being formed by simple juxtaposition of crystals of the two component metals, have a degree of hardness which is intermediate between the hardness of both metals. According to some well known investigations of Grace-Calver the hardness of the alloy can be calculated by the law of mixture.

B. It is also known that brass rich in zinc and that bronze are quite unalterable. This property evidently results from the formation of definite compounds, which must have evolved some heat while being formed, and lost thereby a portion of their power to lend themselves to further chemical reaction.

C. The enormous increase in electrical resistance resulting from the addition of small quantities of certain metals to another pure metal is well known, but the mechanism of this influence is difficult to ascertain owing to our limited knowledge of the true chemical constitution of such alloys. Manganese and nickel, which greatly increase the electrical resistance of iron, form undoubtedly with this metal isomorphous mixtures, but is this the case in all similar instances, in alloys of gold and silver, for example, whose electrical resistance is also very great? It seems probable, but it is not certain.

D. When alloys are made up by the juxtaposition of crystals of the component metals, without the formation of chemical com-



pounds, their fusibility is greatly superior to the average fusibility of the associated metals. In the case of isomorphous mixture, on the contrary, the fusibility approaches the average fusibility.

When a definite compound is formed in the alloy, fusibility is generally greatly decreased, and the melting-point of the combination is then sometimes decidedly superior to that of the component metals.

The influence of the physical constitution, or, in other words, of the structure, upon the properties is not less marked. It is possible to judge of this influence by the examination of the fracture, but only to a very limited extent, especially with regard to the mechanical properties, the malleability for instance. It appears to be well proved that a characteristic relation exists between the grain of the fracture and the elongation of pure metals.

The "cutting" (*frottement*) of bearing alloys appears also to depend upon the structure.

## II

It is evident from these considerations that the first aim of a general study of alloys should be to ascertain accurately their chemical constitution and their structure, and that its final aim should be the study of the relation existing between their useful properties and the constitution previously determined.

If the investigations, carried on in recent years concerning the chemical constitution of alloys, have shown the efficiency of certain experimental methods, which can be now used more generally and systematically, they have also shown that none of these methods is alone sufficient to solve the problem. They need their mutual assistance, because each of them fails in certain special cases, and especially because they are indirect methods, whose results may lead to different interpretations, sometimes to contradictory ones. The measurement of electrical conductivity, for instance, had led to infer the existence of certain definite compounds and of allotropic transformations which were not confirmed by subsequent experiments. In order to avoid such errors, it is necessary to employ simultaneously the various methods which appear to be better adapted to throw some light upon the constitution of alloys, as, for instance, the chemical separation of the compounds, the determination of the fusibility, of the electromotive force and of the electrical conductivity.

The chemical separation of the compounds may be applied to those alloys which contain an excess of the metal more readily acted upon by dissolving them in a suitable reagent. It is possible by this method to ascertain the existence or absence of definite compounds, and, if the alloys be ternary ones, what are the metals which form combinations. Incidentally it may give a means to determine the principal specific properties of the compounds detected.

The fusibility method is based on the fact, ascertained by experiment, that a molten mixture of several metals, when allowed to cool, does not become entirely solid at a constant temperature as is the case with a pure metal. The solidification of such mixture begins at a certain temperature which depends upon the composition of the mass; it progresses as the temperature is further lowered, and is completed at a certain constant temperature. The point at which solidification begins is the one that should be considered as the true solidification point of the alloy; it corresponds to the point of crystallization of aqueous solutions, and the curves representing the relation between these points and the corresponding compositions of the alloys are exactly similar to solubility curves.

The general appearance of fusibility curves may give some very valuable indications concerning the chemical constitution of alloys. It is well known that they are composed of as many branches forming sharp angles, as there are chemically different constituents (definite compounds or allotropic varieties) deposited upon solidification.

The determination of the electromotive forces of solution is of all the methods previously used, the one which seems destined to furnish the most accurate data concerning the composition of the definite compounds present in alloys. When the composition of an alloy of two metals is progressively altered, an abrupt and sometimes enormous variation in the electromotive force of solution takes place when the composition of a definite compound is reached.

The electrical conductivity of alloys may also give some indications concerning the definite compounds, especially in the case of isomorphous mixtures. The enormous decrease of conductivity resulting in a metal from the addition of small quantities of foreign elements, appears to be related to the formation of



isomorphous mixtures. It is, at any rate, the case with alloys of iron and nickel or manganese, and gold and silver alloys.

The existence of direct relations between the physical structure of a metal and its mechanical properties is a fact too well known to be insisted upon.

In all metallurgical works the operations are controlled according to the appearance of fractures whose character depends primarily upon the physical structure and, to a certain extent, upon the hardness and malleability of the elementary constituents.

The variations in the appearance of the fractures of the same metal give very valuable indications concerning its mechanical properties. Unfortunately the appearance of fractures does not admit of any accurate definition; they cannot be permanently reproduced on paper, and do not lend themselves to really scientific researches. Their use must be classed among these empirical methods, undoubtedly very useful in industrial operations, but which it would be desirable to control by some more scientific testing methods, whose employ would only be intermittent.

Metallography created by Sorby and developed by Martens, Osmond and others, fulfils precisely the desired conditions. It reveals the structure itself, that is, the form of the constituents whose juxtaposition constitutes the metallic mass and makes it possible to preserve, through photography, a permanent record of the structure.

The general principle of the microscopical examination of the structure of metals consists in preparing a plane and polished surface by ordinary polishing methods, and to cause the apparition upon this surface of the outlines of the different constituents, in utilizing their different resistance to properly selected reagents.

The investigations just outlined are probably of such character as to yield a sufficiently clear understanding of the physical and chemical constitution of alloys, but they do not reveal the relations existing between these constitutions and the useful properties of the alloys, such as their hardness and malleability, of importance in the mechanical working of metals; their fusibility and electrical conductivity, of importance, the former in castings, the latter in electrical work; their unalterability when exposed to atmospheric agencies, of importance for all metals.

Of these various properties, the mechanical properties alone

demand a separate study. Fusibility and electrical conductivity will have been studied in connection with researches relating to the chemical constituents, while the degree of unalterability will be indirectly ascertained through the measurement of the electromotive force and through the etchings with various reagents called for by the study of the physical structure.

The temptation might be at first to limit the study of the mechanical properties to those alloys which are more likely to be found useful in the arts, to the exclusion of the numerous alloys which owing to their brittleness have no industrial importance. Such course, however, would undoubtedly be a wrong one, for the real value of such investigations consists in bringing to light certain general laws which will manifest themselves much more clearly in the case of extended series of experiments including numerous alloys. It should be noted here that the sterility of many of the researches, already so numerous, dealing with iron and steel, comes from the fact that they were almost always limited to industrial products.

This necessity of studying complete series of alloys, a number of which are extremely brittle, makes it necessary to replace the ordinary tension tests by compression and bending tests. It may be stated with regard to the latter that they yield indications nearly equivalent to those of tension tests and that the amount of deflection is as satisfactory to judge of the malleability of the metal as the elongation.

Ordinary tension tests, moreover, may be made in the case of particularly interesting alloys.

In the investigations so far undertaken, all proportions of the constituent metals have been considered, from 0 to 100 per cent, no special attention having been paid, *a priori*, to any one proportion. It is, however, important to consider separately the case of alloys in which one of the component metals is present only in very small proportion, 1 per cent or less, for instance. These alloys deserve special attention for three reasons:

1. These slight amounts of some elements, added to a metal and generally called impurities, may exert an enormous influence upon certain properties, especially upon the physical structure, the malleability and the electrical conductivity.

2. It is impossible in the production of metals and alloys to completely avoid the presence of some of these impurities.



3. The experimental study of such alloys is accompanied by exceptional difficulties both with regard to their preparation and their chemical analysis.

### III

Such is, briefly outlined, the program of the researches on alloys which the Committee of the Société d'Encouragement deemed important to undertake. Before making a start, however, the Committee thought it advisable to select the alloys which should be first investigated.

From a purely speculative point of view, the alloys to be selected were evidently those composed of metals which could be readily alloyed in all proportions, that is, the alloys of miscible metals, fusible, not easily oxidized when exposed to the air and belonging to various groups, so as not to overlook some interesting phenomena which might belong only to some alloys.

From the exclusive point of view of commercial application, on the contrary, the alloys to select were those in general use, that is, those that could be produced at low cost, and among them preference was to be given to the metals whose cost of production had in recent years been greatly reduced, such as aluminium, nickel, silicon, and which having not yet been subjected to many inquiries, were much likely to yield new and interesting results.

In order to conciliate both views, the Committee was led to divide in two groups the alloys to be studied: one group comprising alloys whose complete study should be undertaken, when mixed in all proportions, from 0 to 100 per cent, and which included the following alloys:

Aluminium-copper, aluminium-nickel, aluminium-zinc;

Copper-antimony, copper-nickel, copper-lead;

Tin-antimony, tin-nickel, tin-zinc;

Antimony-lead, antimony-zinc, antimony-nickel.

The other group included those alloys which called for a partial study only, either because they could not be prepared in all proportions, or because they had already been the object of many investigations. They included the following alloys:

Silicon-aluminium, silicon-copper, silicon-zinc, silicon-tin.

Copper-zinc, copper-tin.

The Committee then decided that the following researches should be undertaken first:

*Chemistry of alloys.*—Determination and study of the definite compounds existing in alloys.

*Mechanical properties of alloys.*—Tenacity, hardness, brittleness, malleability, effect of cold working, hardening and annealing.

*Electrical properties of alloys.*—Electrical conductivity and electromotive force of solution.

*Fusibility of alloys.*

*Influence of impurities in commercial metals and alloys.*

*Magnetic properties of iron alloys.*—This study to be conducted from the point of view of the manufacture of permanent magnets.

In order to fill this broad program, the Committee secured the assistance of scientists and engineers, who had, by their previous work, proved their unquestionable competency in the subjects which they agreed to investigate. It will suffice to mention the names of Mrs. Sklodowska-Curie and of Messrs. Charpy and Gautier, whose important memoirs have been published in the *Bulletin*. For the convenience of the persons who will have to consult them, they are to-day published together in a single volume and, to add to the value of the publication, some personal publications of Messrs. Osmond, Carnot, Guillaume and H. Le Chatelier, on similar subjects, have also been reproduced. These memoirs had already been published in the *Bulletin* or are due to members of the Council.

An important part of the material needed for these researches was generously provided by industrial works and by individuals interested in the success of our enterprise. Among these we find the following names: Mr. Solvay, Société Royale asturienne, the six great French Railroad Companies, the Société des Forges et Aciéries de la Marine, the Chatillon and Commentry Co., the French Metal Company.

The Committee take this occasion to again thank them very warmly for their generosity in favor of an undertaking which, it is to be hoped, will soon prove its industrial importance.

The Committee also desire to express their gratitude to their secretary, Mr. H. Le Chatelier, the active promoter of their labor, to which he has rendered, with absolute devotion, the most precious assistance.



**The Temperature of Rolling.** — To the most advanced American students of the structure and treatment of steel there is scarcely anything new in the abstract we print on another page of Mr. Ridsdale's paper before the Iron and Steel Institute on the correct treatment of steel, but we venture to say that the knowledge acquired by advanced investigators as to the proper treatment of steel in rolling and annealing has, on account of the extremely recent date of the investigations, not yet reached the bulk of those who are connected with the manipulation of steel.

It has been established that more depends upon the proper heat treatment of steel after the ingot has been cast and bloomed than upon either its chemical composition or the process by which it has been made.

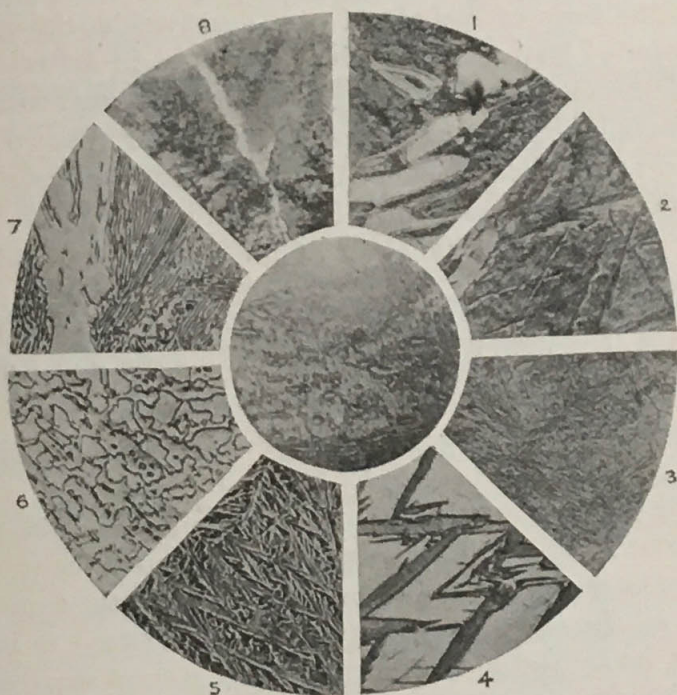
A large percentage of the steel which is being turned out of finishing mills to-day is subjected to treatment which makes poorer material than would result from the employment of steel of analyses which no manager would accept from the steel maker. A single finishing pass at an improper temperature may be worse for the steel than a variation of many points in some of the impurities.

The impression has grown, because until very recently there has been little to controvert it, that when steel is made that is practically the end of it — that heating and rolling concern only the shape. If variations in the physical characteristics cannot be traced back to the chemical composition or method of manufacture it is because the chemist cannot find out all that chemistry should show. Such assumptions are entirely erroneous, and the application of the truths discovered by investigating the changes in the structure of steel caused by heating and rolling will clear up many mysteries and result in the production of much better finished material. — *Tin and Terne*, September 19, 1901.

**Metallography at the National Physical Laboratory.\*** — Let us turn now to the other side, to some of the problems which remain unsolved, to the work which our laboratory is to do and by doing which it will realize the aim of its founders. The microscopic examination of metals was begun by Sorby in 1864. Since that date many distinguished experimenters, Andrews, Arnold, Ewing, Martens, Osmond, Roberts-Austen, Stead and others

\* Extract from a discourse delivered at the Royal Institution by R. T. Glazebrook, F. R. S., Director of the National Physical Laboratory.

have added much to our knowledge. I am indebted to Sir W. Roberts-Austen for the slides which I am about to show you to illustrate some of the points arrived at. Professor Ewing a year ago laid before the Royal Institution the results of the experiment of Mr. Rosenhaim and himself. This microscopic work has revealed to us the fact that steel must be regarded as a crystallized igneous rock. Moreover, it is capable at temperatures far below its melting point of altering its structure completely, and its mechanical and magnetic properties are intimately related to its structure. The chemical constitution of the steel may be



unaltered, the amounts of carbon, silicon, manganese, etc., in the different forms remain the same, but the structure changes, and with it the properties of the steel. The accompanying illustration represents sections of the same steel polished and etched after various treatments. The steel is a highly carbonized form, containing 1.5 per cent of carbon. If it be cooled down from the liquid state, the temperature being read by the deflection of a galvanometer needle in circuit with a thermopile, the galvanometer shows a slowly falling temperature till we reach  $1380^{\circ}\text{C}.$ , when solidification takes place. The changes which now go on take place in solid metal. After a time the temperature again



falls until we reach  $680^{\circ}$ , when there is an evolution of heat; had the steel been free from carbon there would have been evolution of heat at  $895^{\circ}$  and again at  $766^{\circ}$ . Now throughout the cooling molecular changes are going on in the steel. By quenching the steel suddenly at any given temperature we can check the change and examine microscopically the structure of the steel at the temperature at which it was checked.

In the figure, with the exception of specimen No. 6, the metal has not been heated above  $1,050^{\circ}$ , over  $300^{\circ}$  below its melting point.

At temperatures between about  $900^{\circ}$  and  $1,100^{\circ}$  the carbon exists in the form of carbide of iron dissolved in the iron, at a temperature of  $890^{\circ}$  the iron which can exist in different forms as an allotropic substance passes from the *Gamma*-form to the *Beta*-form, and in this form cannot dissolve more than 9 per cent of carbon as carbide. Thus at this temperature a large proportion of the carbon passes out of the solution. At  $680^{\circ}$  the remainder of the carbide falls out of the solution as laminae.

Thus the following temperatures must be noted:  $1,380^{\circ}$ , melting point;  $1,050^{\circ}$ , highest point reached by specimen;  $890^{\circ}$ , 6 per cent of carbon deposited;  $680^{\circ}$ , rest of carbide deposited.

To turn now to the details of the photo, the center piece is the cemented steel as it comes from the furnace after the usual treatment.

These slides are sufficient to call attention to the changes which occur in solid iron, changes whose importance is now beginning to be realized. On viewing them it is a natural question to ask how all the other properties of iron are related to its structure; can we by special treatment produce a steel more suited to the ship-builder, the railway engineer or the dynamo maker than any he now possesses?

These marked effects are connected with variations in the condition of the carbon in the iron; can equally or possibly more marked changes be produced by the introduction of some other elements? Guillaume's nickel steel with its small coefficient of expansion appears to have a future for many purposes; can it by some modification be made still more useful to the engineer?

We owe much to the work of the Alloys Research Committee of the Institution of Mechanical Engineers. Their distinguished chairman takes the view that the work of that committee has only begun and that there is scope for research for a

long time to come at the National Physical Laboratory, and the executive committee have accepted this view by naming as one of the first subjects to be investigated the connection between the magnetic quality and the physical, chemical and electrical properties of iron and its alloys with a view especially to the determination of the conditions for low hysteresis and non-aging properties.

At any rate we may trust that the condition of affairs mentioned by Mr. Hadfield in his evidence before Lord Rayleigh's commission which led a user of English steel to specify that before the steel could be accepted it must be stamped at the Reichsanstalt will no longer exist.

**Importance of Annealing Steel Castings.** — The accompanying photomicrographs and corresponding bending tests taken by Mr. C. M. Carr of the Schickle, Harrison & Howard Iron Co., of St. Louis, demonstrate the importance of properly annealing steel castings. The close relation existing between heat treatment on the one hand and microstructure and physical properties on the other, is here so clearly shown as to render any comment superfluous. It is a forcible illustration of the importance of the study of the microstructure in all questions pertaining to heat treatment.

**Metallography at the Technical Schools.** — The School of Mines and Metallurgy of the University of Missouri, located at Rolla, Mo., have installed a very complete equipment for metallographic work under Professor Herman Schulze.

Much activity in metallography is reported from Harvard University, Columbia University and the Massachusetts Institute of Technology. In these three institutions the number of students engaged in metallographic work is such as to tax the present facilities to their utmost.

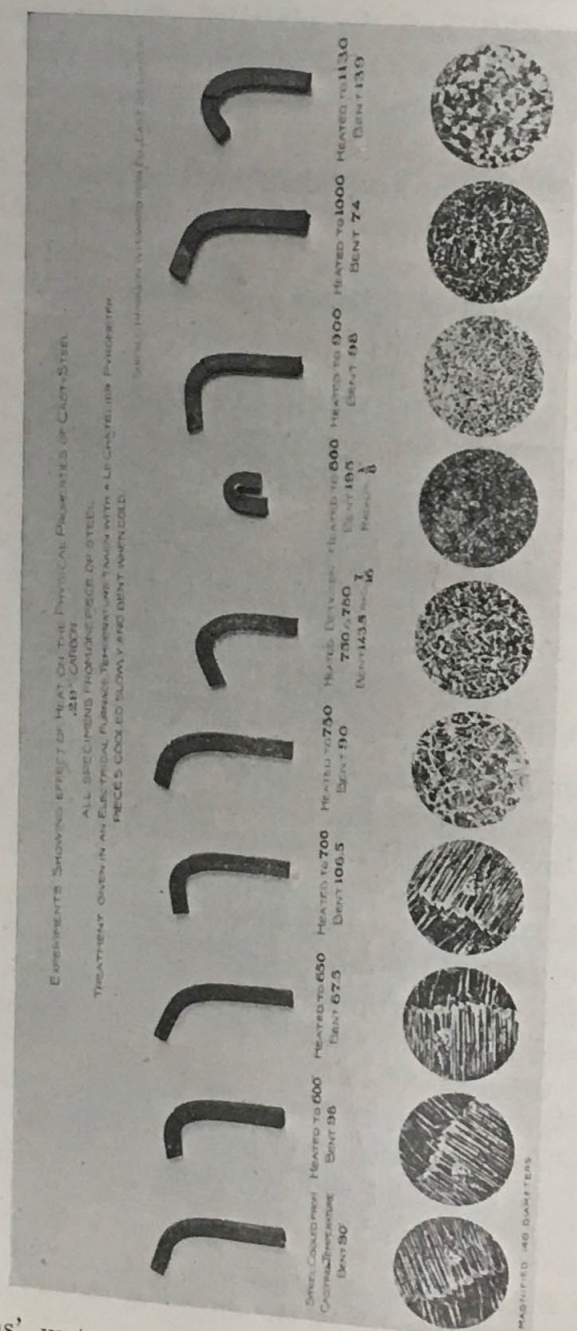
At Harvard University a new Metallographic Laboratory is being equipped. The outfit will be the best and most complete that can be obtained, and will afford facilities for simultaneous work by at least ten students.

**Recent Publications.** — *Metallurgical Laboratory Notes*, by Henry M. Howe, Professor of Metallurgy in Columbia University in the City of New York.

In this book the author describes some sixty-seven laboratory experiments conducted by students in the Department of Metal-



lurgy of the School of Mines of Columbia University on the following subjects: Calibration and use of the Le Chatelier pyrom-



treatment, fracture and micro-structure of steel, Effect of overheating, burning and refining steel, Microscopic metallography (silver-copper alloys and several steels), Tempering of steel, Removal of the effect of cold working by reheating, Hardening as a function of the carbon-content, Calorimetry, Oxidizing and chloridizing roasting of copper sulphide ore, Desilverization of Base Bullion, Shrinkage of refractory materials at high temperature, Seger cones, Melting-points and formation points of various silicates, the action of fluxes on Lead silicates, Properties of Manganese steel, including its heat-treatment, behavior of copper and iron oxides and sulphides on fusion, the copper-selecting process, many experiments connected with the Ziervogel process, the electrolytical refining of copper, the resistance of many kinds of refractory materials to the corrosive action of different slags, the influence of finishing temperature on steel, the prevention of blowholes, and others.

These notes are fully illustrated and include the following very valuable tables which alone are well worth the cost of the book: Synoptical Table for Microscopic Metallography of Carbon Steel — Comparison of Thermometric Scales for every  $10^{\circ}$  C. and every  $25^{\circ}$  F. — Melting and boiling and other important temperatures. — The percentage composition, atomic weights and formulae (with logarithms) of oxides, sulphides, silicates, phosphates, etc.

We reproduce on page 78 the table of thermometric scales.

These notes should prove very valuable not only to the instructors and students of technical schools, but also to Metallurgists and Metallographists engaged in commercial work.

The book is published by the Boston Testing Laboratories. Price, bound in cloth, \$1.00.

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*The Microscope* by Simon Henry Gage, Professor of Microscopy, Histology and Embryology in Cornell University and the New York State Veterinary College. The eighth edition of this excellent treatise on the Microscope has just been published. It is of special interest to the metallographist because it is the first book on the Microscope which alludes to the microscopical examination of photo-micrography of metallic surfaces. The book is published by the Comstock Publishing Co., of Ithaca, New York.



TABLE VI. — *Comparison of Thermometric Scales, for every 10° C. and every 25° F.*

C.	F.	C.	F.	C.	F.	C.	F.	C.	F.	C.	F.
260	500	550	1022	774	1425	1000	1832	1290	2354	1580	2875
270	518	552	1025	779	1434			1300	2372	1580	2876
274	525	560	1040	780	1436	1010	1850			1590	2894
280	536	566	1050	788	1450	1020	1868	1302	2375	1593	2900
288	550	570	1058	790	1454	1024	1875	1310	2390	1600	2912
290	554	580	1075	794	1461	1030	1886	1316	2400		
300	572	580	1076	800	1472	1038	1900	1320	2408	1625	2957
		590	1094			1040	1904	1330	2425	1650	3002
302	575	593	1100	802	1475	1050	1922	1330	2436	1675	3047
310	590	600	1112	808	1486	1052	1925	1340	2444	1700	3092
316	600			810	1400	1060	1940	1343	2450	1750	3182
320	608	607	1125	816	1500	1066	1950	1350	2462	1800	3272
330	625	610	1130	820	1508	1070	1958	1357	2475	1850	3362
330	626	620	1148	830	1525	1080	1975	1360	2480	1900	3452
340	644	621	1150	830	1526	1080	1976	1370	2498	1950	3542
343	650	630	1166	840	1544	1090	1994	1371	2500	2000	3632
350	662	635	1175	843	1550	1093	2000	1380	2516	2050	3722
357	675	640	1184	850	1562	1100	2012	1385	2525	2100	3812
360	680	649	1200	851	1564			1390	2534		
370	698	650	1202	857	1575	1107	2025	1399	2550		
371	700	657	1215	860	1580	1110	2030	1400	2552		
380	716	660	1220	866	1591	1120	2048			C.	F.
385	725	663	1225	870	1598	1121	2050	1410	2570	1° =	1.8°
390	734	664	1127	871	1600	1130	2066	1413	2575	2° =	3.6°
399	750	670	1238	880	1616	1135	2075	1420	2588	3° =	5.4°
400	752	677	1250	882	1620	1140	2084	1427	2600	4° =	7.2°
		678	1252	885	1625	1149	2100	1430	2606	5° =	9.0°
410	770	680	1256	890	1634	1150	2102	1440	2624	6° =	10.8°
413	775	690	1274	897	1647	1160	2120	1441	2625	7° =	12.6°
420	788	691	1275	899	1650	1163	2125	1450	2642	8° =	14.4°
427	800	693	1280	900	1652	1170	2138	1455	2650	9° =	16.2°
430	806	700	1292			1177	2150	1460	2660		
440	824			910	1670	1180	2156	1469	2675		
441	825	705	1300	913	1675	1190	2174	1470	2678		
450	842	707	1304	920	1688	1191	2175	1480	2696		
455	850	709	1308	927	1700	1200	2192	1482	2700		
460	860	710	1310	930	1706			1490	2714		
469	875	714	1317	940	1724	1205	2200	1496	2725		
470	878	719	1325	941	1725	1210	2210	1500	2732		
480	896	720	1328	950	1742	1219	2225				
482	900	722	1332	952	1746	1220	2228	1510	2750		
490	914	726	1338	955	1750	1230	2250	1520	2768		
496	925	730	1346	960	1760	1232	2250	1524	2775		
500	932	732	1350	969	1775	1240	2264	1530	2786		
		736	1357	970	1778	1246	2275	1536	2800		
510	950	737	1359	973	1783	1250	2282	1540	2804		
520	968	746	1364	980	1796	1260	2300	1550	2822		
524	975	750	1375	982	1800	1270	2318	1552	2825		
530	986	756	1382	984	1803	1274	2325	1560	2840		
538	1000	760	1400	990	1814	1280	2336	1566	2850		
540	1004	770	1418	996	1825	1288	2350	1570	2858		

Formulae for converting from one scale into the other.

$$F = \frac{9C}{5} + 32;$$

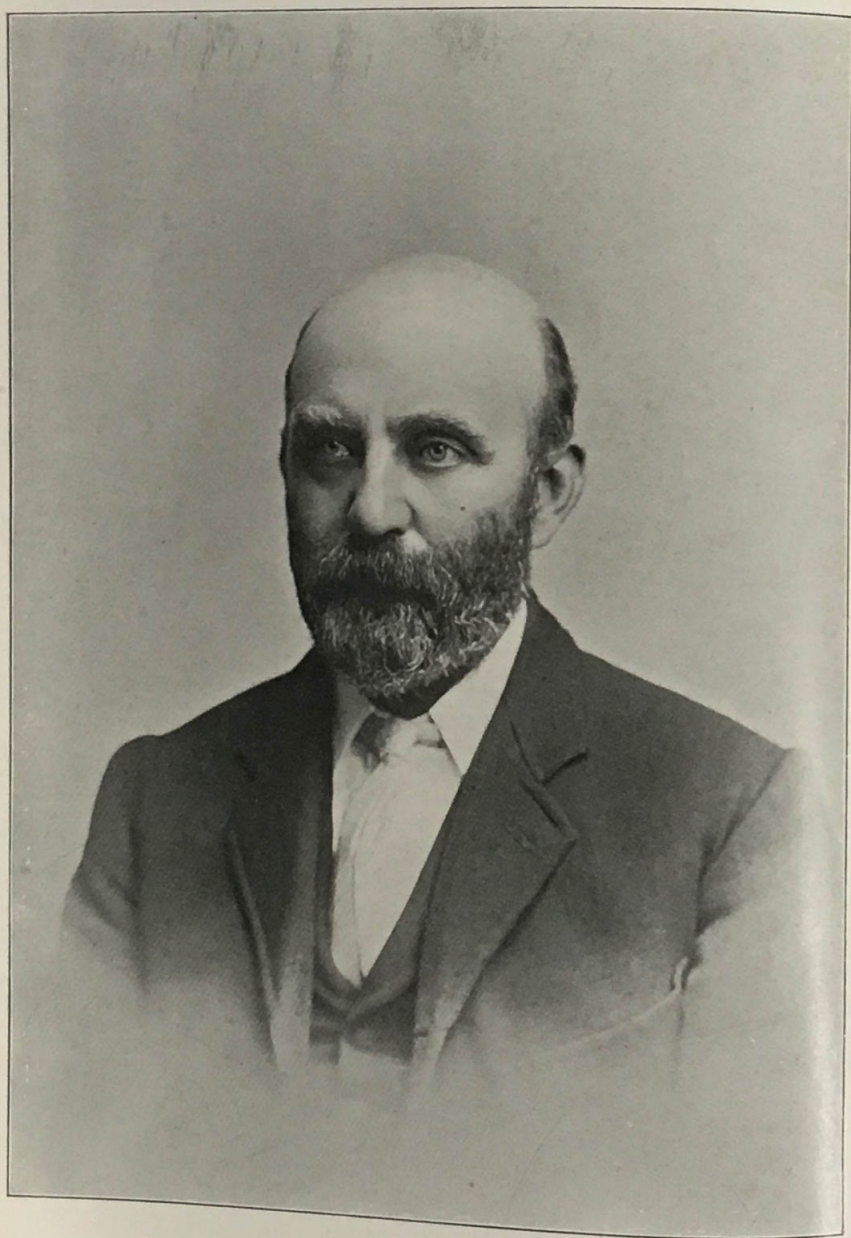
$$C = \frac{5(F - 32)}{9}$$

F = number of degrees Fahrenheit.

C = number of degrees Centigrade.







JOHN E. STEAD

# The Metallographist

A QUARTERLY PUBLICATION DEVOTED TO THE STUDY OF METALS, WITH  
SPECIAL REFERENCE TO THEIR PHYSICS AND MICROSTRUCTURE,  
THEIR INDUSTRIAL TREATMENT AND APPLICATIONS.

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Vol. V

APRIL 1902

No. 2

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## JOHN E. STEAD

THE readers of this journal are too well acquainted with the character and importance of Mr. John E. Stead's work to require more than a short review on the part of the writer of this sketch. Mr. Stead belongs to a very distinguished family, his brothers being W. T. Stead of journalistic fame and the Rev. F. Herbert Stead, warden of the Browning Hall Settlement, Walworth.

The subject of this sketch studied analytical chemistry under his present partner, Mr. Pattinson, at New Castle on Tyne. He was then appointed chemist to the Hebron works of the Thorsis Sulphur and Copper Company, which he left after a short stay to become chemist of the Messrs. Bolckow, Vaughan & Co. In this position he had excellent opportunities to become thoroughly acquainted with iron and steel making.

In 1876 Mr. Stead entered into partnership with his first employer, Mr. Pattinson, at Middlesbrough, as analytical chemist, under the firm name of Pattinson & Stead. His business career has been most successful and his high standing has attracted to his laboratory numerous pupils, some of whom are to be found to-day in many of the iron and steel works of Great Britain.

Mr. Stead holds the public appointments of Borough Analyst, of Analyst under the Fertilizers and Feeding Stuffs act, and of Chemist to the Cleveland Agricultural Society. In 1901 he



was awarded the Bessemer gold medal by the Iron and Steel Institute, of which he has been for many years a member of council.

Mr. Stead became interested in metallographic research soon after the International Engineering Congress, held in Chicago in 1893, and at which several important papers on the subject were presented. Since then much of his energy and exceptional working power have been expended in that direction. His contributions to metallography have been numerous and important. He has devised many little appliances useful to metallographists, and has helped in reducing the time previously required for the polishing of metallic sections. The heat-tinting method first proposed by Martens, was developed by him to a high degree of perfection and applied with much success to the development of the structure of cast iron, more especially for the purpose of revealing the location of the phosphide-holding areas, which, by other methods, are readily confounded with pearlite areas.

Mr. Stead's masterly papers dealing with the crystalline structure of iron and the brittleness caused in soft steel by annealing have added much to our knowledge of the constitution and characteristics of iron and of low carbon steel, while his admirable recent investigations dealing with the detection under the microscope of those constituents in which the phosphorus present in iron and steel, segregate, greatly increases the value of the microscope in the industrial laboratory. His work on the micro-structure and constitution of a number of metallic alloys has materially enlarged our knowledge of these important metallic products.

A complete list of Mr. Stead's papers dealing with metallographic subjects is given below in chronological order:

- Methods of Preparing Polished Surfaces of Iron and Steel for Microscopical Examination. *Iron and Steel Institute*, 1894, Vol. I.
- An Introduction to the Study of Micro-Metallography. *Cleveland Institution of Engineers*, 1895-6.
- Preliminary Micro-chemical Examination of Malleable Castings. *Cleveland Institution of Engineers*, 1895-6.
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## THE CRYSTALLINE STRUCTURE OF METALS \*

By J. A. EWING, and WALTER ROSENHAIN

THE investigations described in the present paper deal principally with the phenomena of annealing. They form a continuation of the research described in the Bakerian lecture for 1899 (*Phil. Trans.*, A, Vol. 193, 1900, pages 353-377).† In iron, steel and brass these phenomena have been studied with the aid of the microscope by various workers, among whom, Arnold, Charpy, Stead, and Roberts-Austen should be particularly mentioned. As a result of their labors it is well known that annealing is accompanied by a re-arrangement of the crystalline grains of the metal.

Thus, when a piece of iron is strained in tension its crystalline grains become elongated in the direction of tension; but

\* *Philosophical Transactions*, Royal Society of London, Vol. 195, page 279. The original paper was illustrated by 37 photo-micrographs, seven of which are reproduced here.—Ed.

† *The Metallographist*, Vol. III, page 94.



when the specimen has been subsequently annealed by being heated to a bright red, all signs of such elongation disappear from the crystalline pattern revealed by the microscope. In fact it is not generally possible to find any definite connection between the crystalline pattern seen in the same specimen before and after annealing. In general, the pattern seen after annealing resembles that found in a similar specimen before it has been strained, but the scale and character of the pattern produced depend very much on the details of the annealing process, particularly upon the temperature applied, the time of its application, and the rate of cooling. Arnold and Stead have shown that prolonged annealing tends to produce large crystals in iron and steel. But even short exposure to a suitable temperature is well known to produce complete re-crystallization, and it has been suggested that these changes occur at critical points corresponding to the "arrest-points" in the cooling of the metal. These arrest-points indicate evolutions of heat, and it is natural to suppose that they are evidences of re-arrangement of the structure of the metal.

At the outset of the present research we hoped to observe this change taking place under the microscope. Although the experimental difficulties of keeping a specimen under microscopic observation while it was being heated were successfully overcome, the attempt to watch the re-crystallisation of iron failed.

Our first expedient was to surround the specimen with an atmosphere of pure dry hydrogen gas, and to examine the surface through a glass or mica window in the containing vessel. The hydrogen was prepared in the usual way by the action of pure dilute sulphuric acid on pure granulated zinc, and the gas was dried and purified by bubbling through several wash-bottles containing pure sulphuric acid, caustic potash, and permanganate of potash solutions respectively. As a final precaution, it was passed through a long tube of glazed porcelain kept at a red heat and packed with clean pieces of sheet-iron. It was a specimen of similar iron that was to be experimented upon, and we hoped that after passing over this large quantity of hot iron, the hydrogen would be free from any impurities capable of tarnishing the polished surface of our specimen. The specimen itself was a strip of sheet-iron, of the pure kind used for transformer plates, measuring 3 centims. in length and 3 to 4 millims. in width. It

was bent to a  $\square$  shape, and the upper flat surface was polished and etched. The containing vessel is shown in section in Fig. 1. It consisted of a brass cylinder, A, with a screw-cap, B, provided with a window of thin glass or mica. The bottom of the cylinder was formed by a plug of slate, C, fitting against a shoulder, and held in place by a screw-collar, D. Through this slate plug passed two stout copper wires, EE; the upper ends of these rods were split to receive the two legs of the specimen F, whose flat, polished surface thus came immediately beneath the window in the cap. At GG tubes entered the vessel; these allowed the passage of the current of hydrogen.

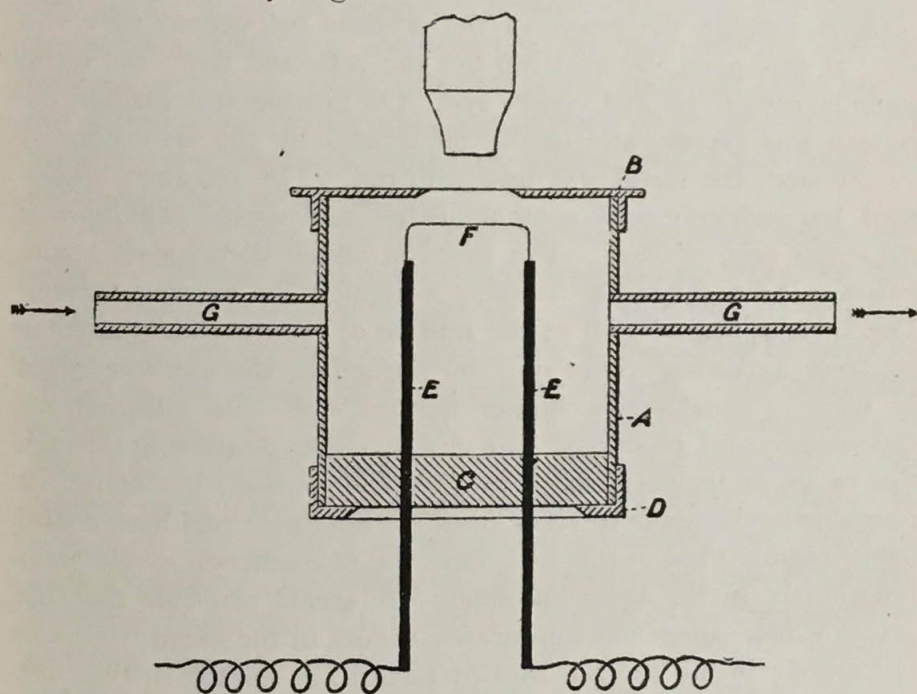


Fig. 1.

All the joints were very carefully fitted, and made as nearly air-tight as possible; leakage, however, was not an important matter as the hydrogen was kept at a pressure slightly higher than that of the atmosphere. On leaving the apparatus the hydrogen was burnt as a small jet.

The specimens were observed through the window by means of "vertical" illumination coming through the objective itself; as we were content with moderate magnifications (up to 100 diameters) an objective of long focus could be used.



In spite of the precautions taken, we did not succeed in keeping the polished surface untarnished after a red heat had been reached; but in the course of our observations an interesting phenomenon was observed. On beginning an observation, when the specimen was still cold, the "ferrite" grains could be clearly distinguished. If the temperature of the specimen were then slowly raised by gradually turning on an alternating electric current, the first visible change was a dimming of the image, which resulted in its becoming completely blotted out. This we supposed to be due to condensation of moisture on some part of the optical system, but we could not locate it. On raising the temperature further, the image of the crystals reappeared very vividly; at this point, by shutting off the reflected light, the metal could be seen to be just vividly red. On heating still further, the pattern was rapidly and totally obliterated by the tarnishing of the surface; the metal was now dull-red. The red then brightened, but suddenly dark spots appeared, and spread rapidly over the entire field of view. The speed at which they spread could, however, be regulated by suitably adjusting the heating current. The spots appeared well in the middle of the specimen, and the apparent darkening could only be pushed to the extreme edges by using a considerably higher temperature. On allowing the specimen to cool, no change was visible, either on passing through this range of temperature or at any other period; nor could the phenomenon be made to recur until the specimen had been cooled below redness; but, if this was done, the phenomenon would recur indefinitely in the same specimen. It seems probable that the action which causes this appearance occurs in the metal itself and not merely in the surface film of tarnish, as details in this film remain entirely unaffected by it, thus giving the observer the impression that he is looking at an action taking place beneath a thin and partially transparent film. On repeating these observations with the specimen maintained in other atmospheres than hydrogen, no such phenomenon was observed; and this leads us to suppose that the phenomenon is a result of chemical reaction between the hydrogen and the iron. From its occurrence just above redness it would seem to correspond to the arrest-point, about  $487^{\circ}$  C., discovered by Sir W. Roberts-Austen ("Alloys Research Report," *Inst. Mechan. Engin.*, 1899).\*

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\* *The Metallographist*, Vol. II, page 186.



Believing that this action of the hydrogen caused the surface of our specimens to tarnish on heating, we sought to overcome this difficulty by observing the surface of a specimen heated in vacuo. The heating was again done electrically, either by passing the current directly through the specimen, or else by placing the specimen in the centre of a coil of fine platinum wire wound on a piece of terra-cotta. In both cases the specimen was placed in a test-tube, the electrodes passing through a sealed cork at the end; the tube could be exhausted by means of a Fleuss pump. The specimen was placed with its polished and etched surface as near to the top of the tube as possible, and a 22 millims. objective was used. When the specimen became really hot, the radiation would have endangered the objective, and this difficulty was met by keeping a blast of air directed partly upon the lower end of the objective and partly upon the surface of the test-tube. The apparatus was thus kept quite cool without in any way disturbing the distinctness of the image.

We found that with the best vacuum we could obtain, even with the addition of phosphoric anhydride drying tubes, our specimens always tarnished if kept at a red heat for any length of time. We are inclined to ascribe this to the action of gases occluded in the metal and given out on heating. The tarnishing, however, was not very rapid, and a specimen could safely be heated up to bright red once before it became noticeable. This enabled us to make observations which very clearly showed that, even with the greatest experimental refinement, we could not hope to observe the process of re-crystallization microscopically. We have found that if a strained, polished and etched specimen, showing distinctly elongated crystals, be gradually heated in vacuo no change whatever is visible in the outlines of the crystals; on removing the specimen from the apparatus it is sometimes found coated with a thin blue tarnish, and on examination with the microscope it is seen that this tarnish has not obliterated the original crystals, but in fact differentiates them by various colors on different crystals. If this tarnish be now polished off, as may easily be done by the use of rouge alone, and the specimen be re-etched, an entirely new set of crystals is revealed. This was readily established by drawing the outlines of the original crystals at a marked spot on the specimen before and after the light re-polishing. Simple re-polishing and etching a similar specimen,



without annealing, produces only very small changes in the pattern. We may therefore conclude that in the above experiment the iron did re-crystallize when at a red heat, but that this re-crystallization did not immediately affect the pattern on the surface. The explanation is obvious when we realize that the pattern seen in the microscope is due to the differences of level and texture in the surface of the specimen which have been produced by the action of the acid used in etching. This pattern, although in its origin dependent upon the actual crystalline structure of the metal at the time when the surface was etched, remains as a mere mask, beneath which re-orientation of crystalline elements may go on without affecting its appearance. The etched pattern is in this respect in the same position as a scratch or mechanical marking of the surface, and the persistence of the etched pattern after annealing is simply an extension into more minute detail of the well-known fact that the external shape of a piece of metal is not in general affected by annealing, although the crystalline structure is entirely changed.

These considerations show that we cannot expect to see the process of re-crystallization in any metal where etching, staining, or relief polishing is needed to differentiate the constituents. We therefore abandoned the attempt to observe this process in iron, but our attention was directed to the study of similar processes of annealing or re-crystallization as seen in more fusible metals, particularly lead.

The question, What is the temperature required to produce re-crystallization in lead? was raised by the appearance we observed in specimens of plumbers' sheet-lead that had been etched with dilute nitric acid. When thus treated, ordinary sheet-lead exhibits a remarkably brilliant crystalline structure on such a large scale that no previous polishing is required. The etched surface shows all the appearances due to the selective effect of oblique light on etched crystalline surfaces; when the specimen is turned the light catches the various crystalline grains in turn, the color and brightness being uniform over each grain, but different on different grains. These surfaces tarnish immediately on drying, and must therefore be observed and photographed while wet with the acid solution.

An examination of this etched surface reveals a peculiarity in the configuration of the crystalline grains; these are seen to



have many remarkably straight boundaries meeting at sharp angles, several sets of parallel boundaries being frequently observed. These features, which strongly resemble what we had previously observed in wrought copper, are to be ascribed to the frequent occurrence of twin crystals. In our earlier observations on twin crystals their presence had always been readily detected by the configuration of slip-bands produced in them by slightly straining the specimen after polishing. An instance of twinning observed in sheet lead by this method has been given by us in a previous paper.\* In the present instance this method of detecting twins is not available, as the roughness of the surface and the great depth of etching employed make it impossible to study the slip-bands. But the presence of twin lamellæ nevertheless becomes evident under slightly higher magnification with oblique light. The selective effect of oblique illumination, picks out a few isolated crystals, lighting these brilliantly while neighboring ones remain almost dark. Within the area of the brightly-illuminated grains, a number of dark patches are seen, and these show the straight boundaries occurring in parallel sets which are characteristic of twinning. As the specimen is turned, the grains that were bright become dark, but presently some of the patches that were previously dark shine out brilliantly, all the bands which are parallel to one another flashing out simultaneously. But those parallel bands which catch the light simultaneously are evidently portions of the crystal in all of which the orientation of the elements has been changed by the same amount; in other words, they are twin lamellæ.

Further evidence of the crystalline structure of sheet-lead is found when a slightly higher magnification (100 diameters) is applied. Many of the crystals are then seen to be marked with geometrical figures, most frequently hexagons, but sometimes triangles and rectangles. These figures are similar, and similarly oriented, over the entire area of any one crystal, but they are generally different in shape and orientation on adjacent crystals. In this respect they resemble the geometrical etched pits in iron and geometrical air-bubbles in cadmium described in our previous paper cited above. Their occurrence in cast lead that has been exposed to dilute nitric acid has been described by Pro-

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\* *Loc. cit.*



fessor Miers and Mr. A. Dick in the *Journal of the Mineralogical Society*, April, 1899. Professor Miers measured their crystallographic angles, but does not connect them definitely with the crystallization of the solid metal — and Mr. Dick's view is that they are promiscuously deposited crystals due to electrolytic transfer of lead from the upper to the lower surface of the specimen. Careful microscopic examination shows that in some instances this is the case in sheet lead. Some of the geometrical figures can be seen to be the outlines of deposited, or at all events, projecting crystals; but the remarkable fact remains that even here these crystals are deposited with their elements in the same orientation as those of the crystal upon which they are being deposited. In many cases, however, geometrical markings are found which form the outlines of pits instead of projections. Sometimes these pits occur on the same grain as the deposited crystals, and both then have similar outline and orientation.

The observations here described form a full demonstration of the marked crystalline structure of sheet-lead, but the origin of this structure is not immediately apparent. Sheet-lead is produced by rolling out cast ingots of the metal without the aid of heat. In the sheets we have, therefore, metal which has been subjected to a very great amount of plastic deformation. The view of plastic strain enunciated in our former paper would lead us to suppose that the structure of such violently deformed metal would still be entirely crystalline, but its crystals would show peculiarities of shape corresponding with the nature of the plastic deformation imposed upon the mass of the metal. Thus, in a specimen which had been rolled from a solid lump into a sheet, we should expect to find the crystals thin and flat, but of considerable area parallel to the surface of the sheet. Sheet-lead, however, shows no such feature; on a transverse section of the sheet no flattening of the crystals is seen. This led us to suppose that some process of annealing or re-crystallization had been at work in the metal subsequent to its manufacture, and, as the lead had not been subjected to any thermal treatment, it seemed that re-crystallization must have occurred in it at ordinary atmospheric temperatures. We therefore directed our experiments to test supposition, and generally to investigate the phenomena of annealing or re-crystallization in lead.

The first step was to determine the effect of very severe

strain on the crystalline structure of lead. In such a soft, ductile metal, plastic deformation may be carried to so great an extent that the adaptability of the individual crystals to change their shape by means of slips on cleavage planes may be insufficient. Careful observation of the crystalline structure of a piece of lead under severe compression confirms this view. Up to a certain point the crystals are gradually flattened out in proportion to the flattening of the whole specimen, but when the "flow" becomes considerable it is found that the crystals, already very thin and flat, are driven into and through one another, this process

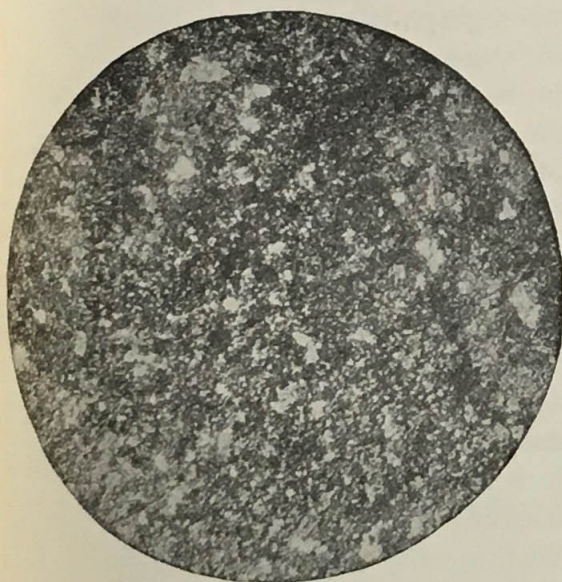


Fig. 2. Cast Lead. Magnified 12 diameters.

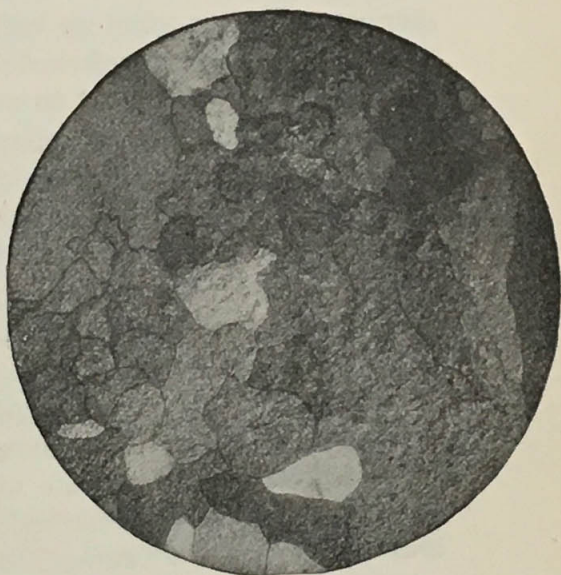


Fig. 3. Freshly and severely strained Lead. Magnified 30 diameters.

resulting in a grain or structure which is small, but still entirely crystalline. The action is analogous to what occurs in the fracture of a more brittle metal, with this difference, that in a more brittle metal, when "slip" has gone so far as to extend right across a crystal, the new surfaces thereby brought into contact do not unite or "weld," and fracture results; in lead the freshly exposed surfaces do weld or re-unite under the pressure, a fact which is associated with the possession, on the part of the metal, of great ductility. Fig. 2 is a micro-photograph showing the crystalline structure of ordinary cast lead magnified 12 diameters, while Fig. 3 shows the much more minute structure of freshly



and severely-strained lead magnified 30 diameters. In this and in the following experiments with lead, the process of straining was carried out by squeezing a cast block of the metal in a compression-testing machine, letting it expand laterally until the block, originally about 1 inch high and  $\frac{5}{8}$  inch diameter was flattened into a plate about  $\frac{1}{8}$  inch thick.

In order to investigate the changes in the crystalline structure of such strained lead, we adopted the method of taking a series of photographs of a marked area of each specimen at intervals of time during which the metal was exposed either to the ordinary temperature of the room or was subjected to special thermal treatment. Before each photograph was taken the surface was thoroughly reetched; our experiments on the annealing of iron had convinced us of the necessity of this proceeding, and our observations on lead specimens have confirmed the previous experience. In no case did thermal treatment in any way produce a visible change in the surface pattern until further etching had been resorted to, and fairly deep etching is required to obliterate the old pattern entirely. This applies more particularly to the channels which are etched out at the inter-crystalline boundaries — these may often be seen forming a network on the newly-formed pattern, but quite independent of the new structure.

The method of etching consisted of alternate applications of concentrated and very dilute nitric acid. In some cases, where very deep etching was required, an electrolytic method was used. One of the great advantages of dealing with a metal like lead arises from the large size of its crystals; by enabling us to use deep etching it allows all fine polishing to be dispensed with, and it becomes possible to obtain photo-micrographs at low magnifications, and under oblique light, which exhibit clearly the various changes in crystalline structure.

The low power required is a further advantage in view of the fact that an etched lead surface must be kept wet with an acid solution while it is being examined and photographed, as such a surface becomes dull and useless as soon as it dries.

To identify the areas which were to be kept under observation, we marked them by scratches or furrows made with a steel point. Unsymmetrical patterns of marking were used for this purpose to facilitate replacing the specimen in its former position and orientation. It is an essential precaution in taking micro-

photographs under oblique illumination which are to serve for purposes of comparison, that the orientation of the specimen and the angle of incidence of the light should be constant throughout the entire series. Our micro-photographic arrangements enabled us to comply with these conditions to a considerable degree of accuracy.

The first series of these photo-micrographs was taken from a specimen of lead which, after being severely strained, was simply kept during six months at the temperature of an ordinary room without any special thermal treatment whatever, being carefully reëtched and photographed at intervals. The first of the series was taken immediately after the specimen had been strained.

The same surface was reëtched and photographed after six days; a small amount of change was visible, and became more marked after one, two, four, and six months respectively. The great change in the crystalline structure was strikingly evident.

Measurement of the largest crystals gives some rough idea of the rate at which these crystals had grown. In one case the dimensions were as follows:

Length of crystal in freshly-strained specimen = .0025"

Length of crystal in specimen after 1 month = .0083"

Length of crystal in specimen after 4 months = .0100"

Length of crystal in specimen after 6 months = .0115"

From this it would appear that the rate of growth is greatest in the first month after the specimen has been strained; but a close examination of such specimens reveals a feature which considerably affects this question. It is seen that the crystals do not grow by the steady accretion of layers all over their surface, but that they throw out arms or branches, which invade neighboring crystals, thus forming a skeleton crystal somewhat similar to those that are often noticed in the crystallization of fused substances.

The figures given above must therefore be taken as no more than an extremely rough indication of the order of magnitude with which we are concerned in these questions of growth at ordinary temperatures.

Taking a specimen of plumbers' sheet-lead showing fairly large crystals as an example of metal in which air-temperature annealing has continued for a long time, we next tried the experi-



ment of applying a higher temperature, so as to determine whether any further change would occur. Our observations show that the metal in this state is very sensitive to moderately high temperatures, three minutes' exposure of ordinary sheet lead to a temperature of  $200^{\circ}$  C. being sufficient to produce a great change in the crystalline pattern. If the specimen be kept at  $200^{\circ}$  C. for a long time there is a continued change, but it becomes very slow,

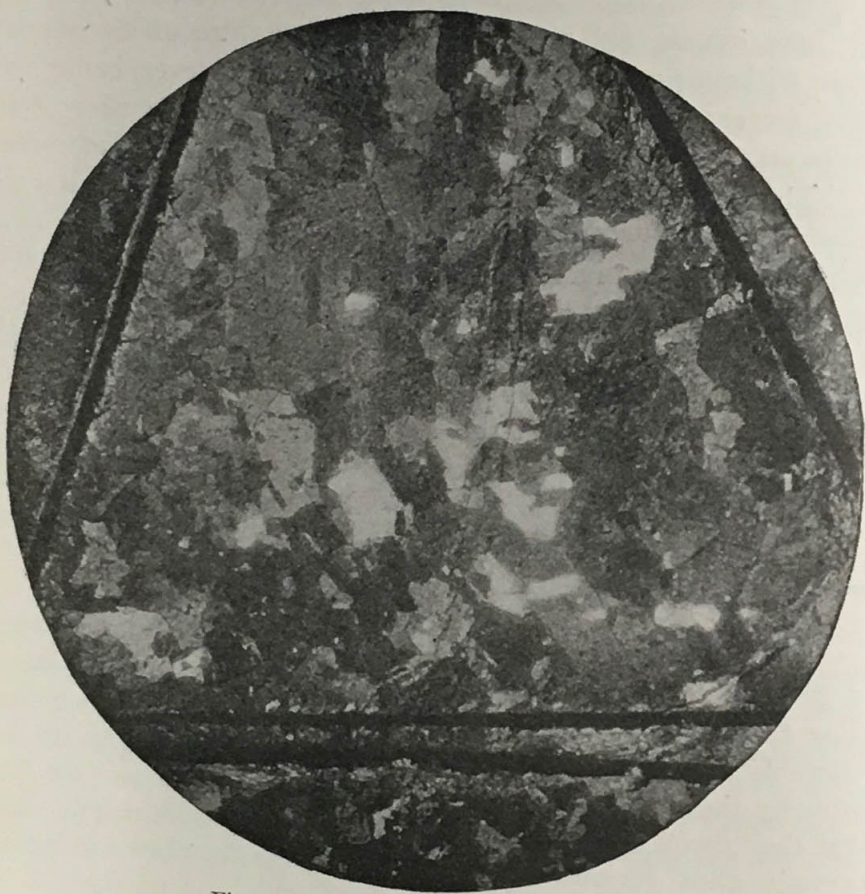


Fig. 4. Typical structure of sheet lead.

and ultimately a state is reached when further change is imperceptible.

Fig. 4 shows the appearance of a typical specimen of sheet-lead, and Fig. 5 shows the change produced by four days at  $200^{\circ}$  C. The latter photograph may be taken as representing the final state of this specimen, as very prolonged annealing produced no further considerable change. This specimen, however, showed

another interesting feature, which we have often observed in other cases. The marked area chosen for photography occupied the centre of the surface of the specimen, which measured approximately  $\frac{3}{4}$  inch square by  $\frac{1}{8}$  inch thick. Somewhat to our annoyance, this marked area did not show by any means the best development of crystalline growth. In this case, as in many others, we found that

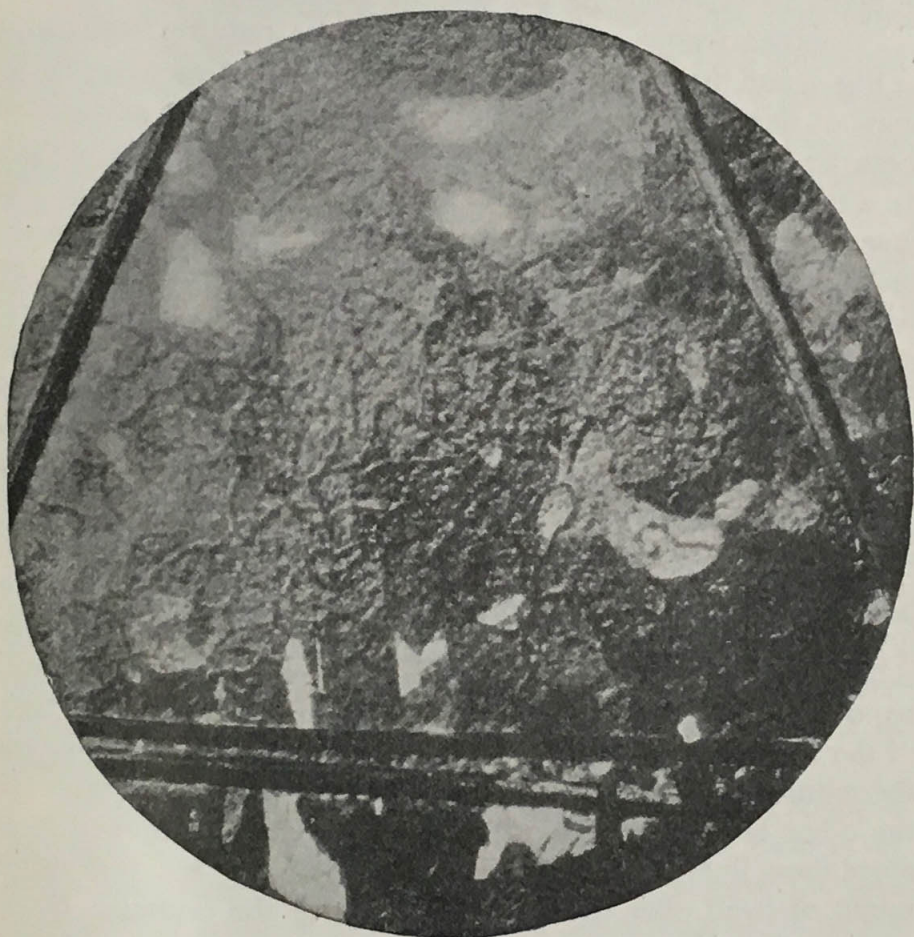


Fig. 5. Sheet Lead kept for 4 days at  $200^{\circ}$  C.

the largest and most rapidly growing crystals were formed at or near the edges of the specimen.

In comparing the members of such a series of photographs, one consideration must be borne in mind, the great difference produced in the appearance of the surface by a minute change in the angle of incidence of the light. In spite of the utmost precaution to keep the incidence constant, a small change will sometimes cause the entire disappearance of a crystal whose facets



happen to be near the critical angle. But, provided any portion of a crystal is shown bright in the photograph, any changes in its outline or surface are certainly genuine, for light that catches the facets on one part of the crystal must necessarily catch all similarly oriented facets also. Where, therefore, the same crystal appears in successive members of a series, the photographs may be taken to represent its life-history accurately.

Another feature of these photographs requires explanation. In Fig. 5 and more or less in most of the others,\* an irregular network of dark lines or channels is seen to cover the surface, and these lines obviously have little or no relation to the crystals. As a matter of fact, these lines are only found in specimens that have been etched, and then annealed and re-etched. When only one etching has been applied, similar lines are seen to follow the inter-crystalline boundaries. In that case they are simply "corrosion channels" caused by the action of the acid at the crystalline junctions. These channels are of comparatively great depth, and when such a specimen is allowed to re-crystallize, and is then re-etched to develop the new crystalline structure, this second etching is rarely carried deep enough to remove the old etched corrosion channels, and these remain as a network over the surface, simply indicating the positions in which crystalline boundaries formerly existed before the structure became changed. This view is borne out by the observation that deeper etching will eventually remove all trace of this network. In many instances these traces of the former crystalline boundaries are very useful as indicating the nature and extent of the growth that has taken place. On the other hand, the deep etching that would be required to remove these lines was considered undesirable, as very deep etching might of itself alter the apparent shape of the crystals. By observing the effect of successive deep etchings on the pattern of a stable specimen we satisfied ourselves that it would require exceedingly vigorous and prolonged etching to cause any visible change of pattern, and that, in the specimens illustrated in this paper, the changes cannot possibly be ascribed to that cause.

Our next experiment was to expose a freshly-crushed specimen of lead to a temperature of  $200^{\circ}$  C. for a long time, and to watch the changes produced in its crystalline structure. The an-

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\* Not reproduced here. — Ed.

nealing ovens\* used for this purpose consisted of wooden boxes, lined with asbestos cloth, and heated by means of one or more incandescent electric lamps. The temperature could be regulated by regulating the current in the lamps, and, more conveniently, by adjusting the lids of the boxes so as to admit more or less air. Variations of  $10^{\circ}$  C. in the temperature were rare.

Photographs were taken after the specimen had been exposed to  $200^{\circ}$  C. for periods of time varying between 17.5 hours and 39 days, 20 hours. They exhibited a steady growth of the crystals.

The occurrence of large crystals in the annealed metal is apparently in no way dependent upon the size of the crystals in the original state before straining.

A remarkable feature of these large crystals is the frequent occurrence of twin crystals, both as inclusions in the body of the crystal and at the boundaries.

In our previous paper we have already given the observation that twin crystals are rarely, if ever, found in a *cast* metal, but that they are frequently developed by strain, and that they are very commonly found in metal that has been annealed after severe straining. Our present observations on lead entirely agree with these statements; but it may now be possible to suggest an explanation for the frequent occurrence of twins in annealed lead. When the metal solidifies from the liquid state, it does so by the formation of skeleton crystals, starting from a great number of centres, and the arms of these skeletons continue to grow until arrested by meeting with other growths.

From these arms other arms again shoot out, and so on until the entire metal is solidified; but each crystalline element as it settles into place on any of these arms must assume the proper orientation to enable it to fit in, and in the process of filling space by means of such a system of many meeting and interlacing arms, the formation of a twin would be almost impossible. But when the metal re-crystallizes after severe strain, it does so by the growth of skeleton arms that must often start from a cleavage plane of an actual solid crystal, and probably the new elements deposited upon such a plane would find it as easy to assume the twin orientation as the normal. The idea that twin crystals are

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\* These annealing ovens were devised and used by Mr. S. R. Roget, in his researches on effects of prolonged heating on the magnetic qualities of iron. *Proc. Roy. Soc.*, May 12 and Dec. 8, 1898.



formed in annealed metal by growth starting from cleavage planes which have been sheared across in the process of compression is suggested by the very straight boundaries observed as a characteristic feature of twin lamellæ.

Having observed the comparatively rapid growth of crystals in strained lead at  $200^{\circ}\text{C.}$ , we extended our experiments to both higher and lower temperatures. Our observations were recorded photographically as before, but the general character of the results is so similar to those already illustrated that it is unnecessary to reproduce these photographs. Experiments were made at temperatures of  $100^{\circ}\text{C.}$ ,  $150^{\circ}\text{C.}$ ,  $250^{\circ}\text{C.}$ , and  $300^{\circ}\text{C.}$

In the absence of a reliable method of measuring the rate of growth of crystals, the following statements must be taken as based upon a rough general estimate. With this reservation, our observations over this range of temperatures may be summed up as follows:

(1) In lead which has been severely strained, re-crystallization goes on at all temperatures from that of an ordinary room up to the melting-point.

(2) The higher the temperature the more rapid are the changes in the crystalline structure.

(3) The rate of change varies with different specimens, probably depending upon the nature and quantity of impurities present, and upon the severity of the strain to which the metal has been subjected.

(4) The size of the crystals ultimately produced does not vary appreciably between  $100^{\circ}\text{C.}$  and  $300^{\circ}\text{C.}$  In lead annealed at the temperature of the air, very large crystals have not yet been obtained, but this is probably only a question of time.

(5) The phenomena of growth of crystals occur in lead only when the metal has previously been subjected to severe plastic strain. The structure of a cast specimen remains unaltered at temperatures which cause a strained specimen to show rapid change. By casting in a mould arranged to cause rapid cooling, specimens of lead can be obtained having a minute crystalline structure, whose scale is not very different from that of severely crushed lead; such a specimen was exposed to  $200^{\circ}\text{C.}$  for nearly seven days, but no visible change of structure occurred. A piece of this specimen was then strained by severe crushing, and on further exposure to  $200^{\circ}\text{C.}$  vigorous growth took place.

(6) The rate at which a lead specimen is cooled from temperatures of  $200^{\circ}$  C. to  $300^{\circ}$  C. down to the ordinary air-temperature has no visible effect on the structure. Even "quenching" in liquid air has no visible effect; quenching in water, cooling in air, and slow cooling in the oven, were all tried on a number of specimens without in any way affecting them.

(7) Lead is mechanically hardened to a small extent by severe strain, and the subsequent effect of annealing in restoring softness is correspondingly small. In one of the experiments a specimen of lead was crushed under a given load in the testing-machine, and the load was left on until no further creeping occurred. The specimen was then annealed and again placed under the same load, when a distinct amount of further crushing was seen to take place.

Some of the experiments described above as having been made with lead were extended to certain other metals that lend themselves to similar treatment; those used were tin, cadmium and zinc.

The crystalline structure of tin is well shown when a surface of a cast ingot of the metal is etched with strong hydrochloric acid. These crystals are generally large, but a much more striking display is obtained on etching the surface of commercial tin-plate. Even before etching, the inter-crystalline boundaries may be seen on the surface, where they are marked by fine grooves or channels. The presence of these channels is readily accounted for by the method of manufacture, during which these plates are drawn out of a bath of melted tin, and allowed to drain. As the plate is drawn out, the layer of tin adhering to it crystallizes, but any fusible impurity present in the tin would remain fluid slightly longer, and, being forced by the crystallizing tin into the inter-crystalline junctions, the still fluid impurities will drain off, thus leaving a minute channel.

The appearance of the etched surface of commercial tin-plate is shown in Fig. 6, which is a photograph at one-half the natural size. In this photograph the outlines of the large crystals can be clearly seen, but it also illustrates another and peculiar feature of etched tin-plate. In all cases of an etched crystalline metal viewed by oblique light we have always observed that, under a given incidence of light, certain crystals were bright while others were dark, and that the illumination was uniform over the



entire area of each crystal. In the etched tin-plate this is not the case, the brightness shades off across individual crystals, sometimes uniformly, so as to give the crystal a concave appearance; at other times in patches, giving a mottled effect. On closer inspection, it appears that such variations in brightness occur only in one direction in each crystal; in that direction the brightness may be made to move across the crystal by a slight oscillation of the

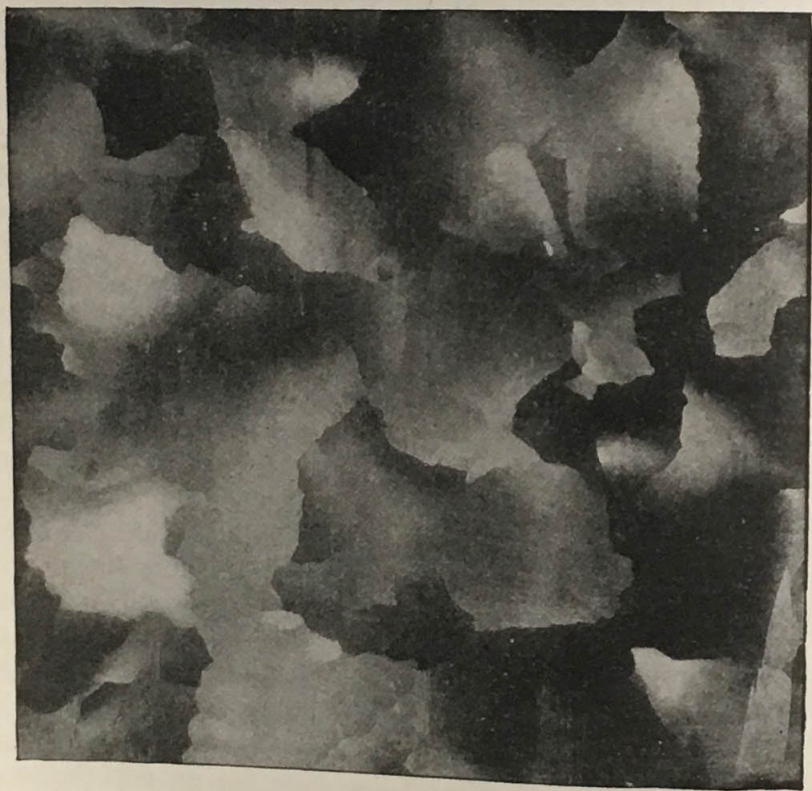


Fig. 6. Commercial tin plate half natural size.

plate, but another direction can generally be found where the crystal appears uniformly bright and is quite free from the concave appearance.

It is well known that the usual selective effect of oblique illumination on etched crystalline surfaces is due to the fact that etching develops on the surfaces of crystals a great number of minute but similarly oriented facets or pits, and the uniformity of illumination over any area is a direct result of the constancy

of orientation of these pits over that area. In the specimens of tin-plate here described we would therefore expect to find that the orientation of the etched pits or facets was not strictly constant over the entire area of each grain.

By examining the etched surface of tin-plate under the higher powers of the microscope the etched pits or facets can be clearly resolved. Under this power their orientation is apparently quite constant, and even under a power of 1000 diameters the departure from strict constancy is only very slight. That it does exist could only be seen by comparing the outlines of apparently similar pits at opposite ends of a large crystal; even there the change in the angles of the figure or of the orientation of one of the sides, as compared by means of a cross wire in the eyepiece, was too small to be measured, but in certain cases the character of one of the outlines of the pit altered slightly, being a fine line at one end of the crystal, and a narrow wedge-shaped dark area at the other. This appearance indicates a change in the slope of that side of the pit, and thus a change of orientation of the internal facets of the pit. This change of orientation seems therefore to occur principally — if not entirely — in the vertical plane, i.e., in the plane of least thickness of the layer of tin.

Under these circumstances it seems that the term "crystal" should only be used with some qualification in speaking of the patches developed by etching the surface of commercial tin-plate. In a crystal which is undistorted by elastic strain constancy of orientation of the elements is an essential characteristic of a true crystal. Some light is thrown on this matter by the fact that we have not observed this "concave" appearance in samples of solid tin, but only on commercial tin-plate where we are dealing with a very thin layer of tin adhering to sheet-iron. Even in tin-plate this effect may be made to disappear by re-melting the tin over a flame and allowing it to cool slowly and at rest.

In view of these observations we are inclined to attribute the "concave" appearance to a distortion or bending of true crystalline plates; such distortion would probably arise from differences in the coefficients of expansion of iron and tin brought into action by a suitable rate of cooling. Considering the extreme thinness of the layer of tin, the amount of distortion might well be purely elastic and insufficient to produce slip in the crystals of tin.



The dimensions and arrangements of the tin crystals are altered by changing the rate of solidification. By means of local quenching and re-melting a great variety of patterns can be obtained; such processes have long been in commercial use in the manufacture of what is called "*moirée métallique*."

It is important to notice that the small crystals of tin which are obtained by quenching the melted metal in water do not show any growth when the metal is exposed for long periods to temperatures short of the melting-point. Even a temperature just short of fusion does not make them grow or re-arrange themselves. A solid block of tin may, however, be reduced to a minutely crystalline structure by severe compression, and in specimens so treated we have observed re-crystallization to occur at  $150^{\circ}$  C.

We also made some experiments on the re-crystallization of cadmium at moderate temperatures. This metal also can be strained by compression until its crystalline structure becomes minute through interpenetration of the original larger crystals. After annealing at  $200^{\circ}$  C. a decided growth of the crystals was observed. Although the gradual growth of some of the crystals is very strikingly shown, however, many of the features that we have observed in the case of lead are entirely absent. In the cadmium we can see no invading branches and no aggressive individuals, nor does there seem to be any considerable amount of twinning.

Experiments similar to those just described were also made on specimens of zinc, with the result that specimens of zinc strained by compression at ordinary temperatures were found to re-crystallize on exposure to  $200^{\circ}$  C. Some results obtained with sheet-zinc, such as that used for electric batteries, were particularly interesting. It is a well known fact that the mechanical properties of zinc are widely different at different temperatures, particularly that the metal is soft and ductile at temperatures slightly above  $100^{\circ}$  C., and that it is generally worked at that temperature, while it is known to become very brittle at and above  $200^{\circ}$  C. Commercial sheet-zinc, rolled at temperatures above  $100^{\circ}$  C., remains fairly soft and flexible at ordinary temperatures, and its crystalline structure is too minute to be seen in specimens etched without previous polishing.

If a specimen of such sheet-zinc be exposed to a temperature

of 200° C. for about half-an-hour, it shows on etching with strong hydrochloric acid a brilliantly crystalline texture. The metal is then much harder, but at the same time brittle; when bent it emits a "cry" like that of tin, and finally breaks with a crystalline fracture which zigzags along the cleavage-planes of the various crystals through which it passes.

Many of the phenomena described above as occurring in such metals as lead, tin, zinc, and cadmium, have close and well-known analogues in iron and steel. In those metals, however, the temperatures involved are much higher, and the experimental difficulties are therefore greater; we have consequently been able to make many observations on the re-crystallization of lead which it has not yet been possible to make in the case of iron or steel. The question, therefore, suggests itself, how far the analogy between the various metals holds true. One view which is held by many persons — perhaps unconsciously — may be called the theory of "corresponding temperatures;" the central idea of this view is that the properties and behavior of metals are to a great extent a function of their distance from their own melting points. Thus a phenomenon seen in lead at 200° C. is strictly analogous to a similar phenomenon seen in iron at 800° C. To a certain extent this view is justified, but it may easily be carried too far. Thus, while lead at 10° C. is soft and ductile like iron at 800° C., yet gold at 10° C. is also soft and ductile, while it is further from its melting-point than iron at the same temperature. Again, one of the most characteristic features of lead is the extreme straightness of its "slip-bands," while those of iron are characteristically curved and irregular. We have not had an opportunity of examining the slip-bands produced by straining red-hot iron, but we have made the converse experiment of straining lead at the temperature of liquid air, with the result that the slip-bands developed were as characteristically straight and regular as ever, thus showing that the straightness of the slip-bands, which is so closely associated with softness and ductility, is a characteristic of the metal rather than a function of the temperature. In order to thoroughly test the same question in relation to annealing, a long series of experiments to determine whether or not gradual re-crystallization goes on in strained iron at ordinary temperatures and a similar series on lead at much lower temperatures would



be required. In the case of glass-hard steels it is well known that a certain amount of annealing takes place at  $100^{\circ}\text{C}$ ., and even at atmospheric temperatures; but in the case of steel, where a distinct chemical change is involved, the nature of the annealing action may differ very considerably from that in an approximately pure metal. Certain phenomena in the recovery of elasticity after over-strain in both iron and steel (Muir, J., "Recovery of Iron and Steel from Over-strain," *Phil. Trans.*, 1899) also point to the fact that changes of internal structure may occur in iron at very moderate temperatures; further evidence in the same direction is afforded by the effect produced (Roget, S. R., *Proc. Roy. Soc.*, 1898) on the magnetic properties of soft iron by roasting at moderate temperatures. On the other hand, it has been very widely believed that annealing or re-crystallization, particularly in iron and steel, are "critical" phenomena which can only occur at or above certain definite temperatures. Arnold has gone so far as to make a careful determination of such an "annealing-point." Various of the "arrest-points" in the cooling of iron and steel have also been regarded as representing critical points in respect to annealing, but the connection between the two is by no means fully proved. Having found phenomena of annealing or re-crystallization in other metals, it therefore became interesting to inquire whether any corresponding arrest-points could be found in the cooling of these metals. We investigated the matter by means of a pyrometric arrangement consisting of two thermo-electric junctions, a very sensitive D'Arsonval galvanometer, and a potentiometer somewhat similar to that used by Sir W. Roberts-Austen; the deflections of the galvanometer were, however, observed by means of a telescope and scale, instead of being photographically recorded. It may be, therefore, that either from this cause, or from insufficient sensitiveness of the whole arrangement, some minute arrest-points were overlooked; but between the melting-points and the ordinary temperature of the air no trace of an arrest-point was observed in the three metals tried, i.e., lead, tin, and cadmium. Such arrest-points, if they exist at all, may be found at much lower temperatures than those to which our experiments were carried.

Having thus failed to correlate the phenomena of re-crystallization in lead, etc., with any definite point at which heat is evolved during the cooling of the metal, and having reason to

believe that even in iron the arrest-points are not necessarily intimately connected with annealing, we look for a theoretical explanation of these actions in another direction. The theory of re-crystallization which we shall now advance as a working hypothesis for the explanation of the phenomena described in this paper ascribes an important part in the action to the impurities present even in "pure" metals. The impurities which we believe to be of importance are those which are capable of forming eutectic alloys, or fusible compounds, with the metal itself; they would therefore be mainly metals, particularly the more fusible metals, such as bismuth, tin, cadmium, mercury, sodium, or even rarer metals, such as gallium. It is well known that when a metal containing a small proportion of such impurities crystallizes, the impurities are, for the most part, segregated in the inter-crystalline boundaries. The crystals themselves form at a temperature when the eutectic alloys are still quite fluid, and the growing crystals gradually push the remaining eutectic into the boundaries. Where the quantity of impurities present is sufficiently great, this eutectic can be seen under the microscope forming an inter-crystalline cement. Mild steel, where the "pearlite" plays the part of a eutectic, is a good example of such a structure; other examples can be found in the gold-aluminium alloys illustrated by Messrs. Heycock and Neville.\* Where the quantity of eutectics present is very small, the meshes of inter-crystalline cement cease to be visible, but the presence of the impurity makes itself felt by the formation of deep grooves or channels along the inter-crystalline boundaries on etching. Even a very minute amount of impurity would suffice to form a thin but practically continuous film of eutectic in the crystalline boundaries.

The close analogy between alloys and salt solutions has been so fully worked out that it is barely an assumption to say that a eutectic alloy in contact with crystals of one of its constituents will behave in much the same way as a saturated solution of a salt in contact with crystals of that salt would do. Our view, then, is that there is constant diffusion from the surface of the crystal into the eutectic film and equally constant re-deposition

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\* "Gold Aluminium Alloys," Heycock and Neville, *Phil. Trans.*, A, Vol. 194, plates 4 and 5.



of metal upon the crystal from the eutectic film. If there are several crystals in contact with the same eutectic, then there will be, under some conditions, a state of dynamic equilibrium between them, the amount dissolved from each being exactly counterbalanced by the amount deposited upon it; if, however, there is any difference in the solubility of various crystals, in other words, if there is any difference in their "solution pressure" in respect to the eutectic, then the less soluble will grow at the expense of the more soluble. In the case of salt crystals in an ordinary solution it is well known that the large crystals gradually absorb the small ones, a transformation generally explained on the ground that a system tends to assume a position of minimum potential energy. In the case of metallic crystals in a solid metal we have, however, this special condition, that the eutectic (or solution) exists as a mere thin film in contact with only one face of one crystal on either side. If, therefore, these two crystal faces differ in solubility or "solution pressure" in the eutectic, gradual transfer of the metal by diffusion through the eutectic film from one crystal to the other would result, and the one crystal would grow at the expense of the other. This action might go on while the eutectic was solid—Sir W. C. Roberts-Austen has demonstrated that diffusion does occur in solid metals,\* but at extremely slow rates, unless the metals are near their melting-points. The metal constituting the eutectic films, being much nearer its melting-point than the rest of the mass, would thus be favorable to comparatively rapid diffusion, but the rate of such diffusion and, consequently, the rate of growth of crystals, would be enormously increased by heating the metal to a temperature above the melting-point of the eutectic in question.

The theory† which we suggest to explain crystal growth in a solid metal depends upon the existence of a difference in the solubility of the two crystal faces in contact with a eutectic film. The only difference between these two faces is, apparently, in the orientation of the crystalline elements; but this very dif-

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\* Bakerian Lecture, "Diffusion of Metals," by W. C. Roberts-Austen, *Phil. Trans.*, A, 1896, Vol. 187, pages 383-416; "Diffusion of Gold into Lead," *Proc. Roy. Soc.*, May 5, 1900.

† It is proper to say that the credit for this theory belongs to Mr. Rosenhain.—J. A. E.

ference is sufficient to produce a difference in the rate of solution of such surfaces in an acid. A polished surface of metal when attacked by an acid is not attacked at a uniform rate all over, but at different rates over different crystals, in such a way as to produce marked differences of level between adjacent crystals. Another phenomenon, seen best in etching lead in dilute nitric acid, is also of interest in this connection; it has already been described in the present paper. We there have a case of lead dissolved from one crystal and deposited upon another crystal in its proper orientation.

In view of these facts, we think it must be admitted that different crystal faces, having a different orientation of their elements, differ in solubility in the same solvent. To inquire into the cause of this difference is a further step in speculation which is, perhaps, hardly necessary in this connection. Such differential actions may, however, most probably be attributed to differences of electrical potential in the surfaces involved. If we accept this view of the matter, then the diffusion across films of eutectic becomes a case of electrolysis. Now, while diffusion in metals and alloys is a proved fact, the possibility of electrolysis in an alloy has not yet been demonstrated experimentally.\* On the other hand, the close analogy with salt solutions leads one to expect that alloys could be electrolyzed, and those who have experimented in the matter are not by any means certain that greater experimental resources will not enable them to electrolyze alloys.

There is at least one fact in the phenomena of re-crystallization which the solution theory, apart from electrolysis, does not cover, while the electrolytic theory explains it very readily. We refer to the fact that only strained crystals will grow, while unstrained crystals show no tendency to change even at higher temperatures. The explanation, on the electrolytic theory, is that in the unstrained state the crystals are surrounded by prac-

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\*It has, however, been shown by Garnier (*Comptes Rendus de l'Académie des Sciences*, Vol. 116, 1893, pages 144-9), that the diffusion of carbon into iron is affected by the action of an electric current. He interposed a layer of carbon between two iron electrodes enclosed in a fire-clay tube; the whole was heated to  $1000^{\circ}\text{C.}$ , and a current of 55 ampères was passed for three hours, when the anode was found to be unchanged, while the cathode had undergone considerable cementation. This action in the interior of the iron is practically electrolysis of the carbon-iron eutectic.



tically continuous films of eutectic, and that electrolysis only becomes possible when severe distortion has broken through these films in places, allowing the actual crystals to come into contact; the electrolytic circuit would then be for each pair of crystals, from one crystal to the other by direct contact and back through the eutectic film.

Our view of the phenomena of re-crystallization in solid metals may be summed up thus:—We believe that the action is one of solution and diffusion of the pure metal constituting the crystals into the fusible and mobile eutectic forming the inter-crystalline cement. This diffusion results in the growth of one crystal at the expense of the other, owing to differences in solubility of the crystal faces on opposite sides of the eutectic film, and it seems probable that this phenomenon of directed diffusion is really a form of electrolysis.

There is one deduction from this solution theory of re-crystallization that lends itself to experimental investigation. If eutectics play an essential part in re-crystallization, these phenomena should disappear in the total absence of impurities capable of forming eutectics. In a perfectly pure metal, re-crystallization by annealing after severe strain should not occur; but it is almost hopeless to obtain a specimen of such purity as to justify the conclusion experimentally in this form. The degree of purity required can be roughly gauged from the fact that 0.1 per cent of carbon in iron is easily visible as "pearlite" under the microscope; so that, probably, an impurity of one part in one million would vitiate the experiment.

But our conclusion can be narrowed down to more practicable limits by putting it in this way:—That if the presence of a eutectic is essential to crystalline growth, then a crystalline boundary free from eutectic should be a barrier to all such growth. This condition can be approximately realized by means of a weld between two clean-cut surfaces of a metal. Lead lends itself particularly well to such experiments, as it welds readily under pressure without the aid of heat. In this way we have obtained a strikingly experimental verification of the conclusion deduced from the above theory.

The welds were made in various ways; generally two lead discs about  $1\frac{1}{2}$  inch in diameter had their surfaces scraped clean with a clean, sharp knife, the two surfaces being put into contact

immediately after scraping. They were then subjected to a pressure of 5 tons steadily applied in a testing-machine; in some cases pressures up to 50 tons were used. The behavior of the specimens under pressure depended upon the previous preparation of the lead discs; as a rule, and in order to obtain the metal in a condition where its crystals would grow rapidly, these discs were prepared by crushing a cast cylinder an inch long by  $\frac{5}{8}$ -inch diameter. In other cases the discs were obtained by casting, and were then only strained when the welding pressure came upon them; in these cases there was considerable "flow" while the two discs were in contact, but their ultimate behavior was the same in all cases.

The welded discs so obtained were found to be firmly united and could be sawn into sections as desired; they were cut up into sections suitable for microscopic examination, sometimes before, but generally after "annealing." The annealing was done by exposing the specimens to a temperature of  $200^{\circ}$  C. for a considerable time—varying from 24 hours to over a month, and the crystals in all cases grew vigorously. Sections at right angles to the plane of the weld were then cut smooth and etched for examination. On the freshly-cut surface the line of the weld could never be distinguished. The etching had to be carried to a considerable depth, because we found that the cut surface was covered by a thin layer of very minute crystals—evidently the result of the violent strains set up by the cutting-tool.

Microscopic examination, generally at 80 diameters, of these etched sections showed that, although the crystals on either side of the weld had grown vigorously, none of them crossed the line of the weld, which was clearly visible as an inter-crystalline boundary continuous along the whole specimen; in many cases aggressive individual crystals had grown up to the line of the weld and there ended quite abruptly. It must not, however, be supposed that this weld line was mechanically weak; it proved on trial to be quite as difficult to cut or tear the metal along the weld as in any other direction. The weld, therefore, behaved as a true inter-crystalline boundary, differing only in the absence of eutectic, and therefore forming a barrier to crystalline growth. Fig. 7 shows the appearance of such a weld in section after annealing and etching, at a magnification of 30 diameters. The line AB is the weld. As these experiments were made on com-



mercial lead, we were prepared to find that, as a mere matter of probability, some eutectic would have occasionally found its way into the welding surfaces, but this seems to have happened only very rarely. We examined some forty specimens, and only in two instances did we see a slight amount of crystalline growth crossing the line of the weld. We think that we are justified in attributing these rare exceptions to the accidental presence of impurity.

We then went a step further. If we have in a welding surface an inter-crystalline junction which acts as a barrier to

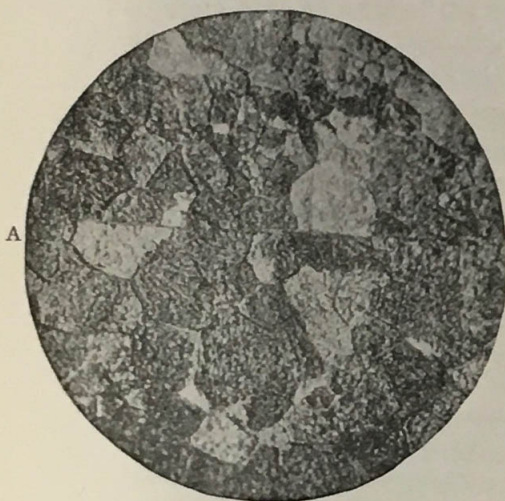


Fig. 7. Cold Weld in Lead, using clean surfaces, after prolonged annealing. Magnified 30 diam.

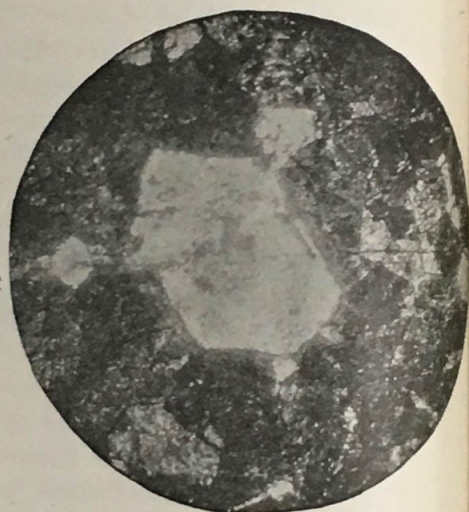


Fig. 8. Cold Weld in Lead, using eutectic in the weld, after prolonged annealing. Magnified 30 diam.

crystalline growth owing to the absence of eutectic, then if a suitable eutectic be supplied, growth should occur there as elsewhere.

Our first experiment was to interpose a thin but continuous layer of lead-bismuth eutectic between the lead discs in welding; the specimen was then annealed for several days at  $200^{\circ}\text{C}$ .—well above the melting-point of the eutectic—but on examination it was found that the layer of eutectic had persisted as such, and allowed no growth to cross it. But in this case the film of eutectic introduced at the weld was continuous, and the conditions were therefore analogous to those which hold at

the boundaries of unstrained crystals, where, as we have pointed out, growth does not occur. To make the experiment conclusive it was necessary to have a discontinuous film of eutectic at the weld. We accordingly tried another experiment, introducing only a few small flakes of the same alloy, and after annealing we found that crystalline growths had crossed the line of the weld in many places. This experiment was repeated many times, various impurities being used, such as the lead-tin-cadmium-bismuth eutectic, pure tin, cadmium, bismuth, and mercury. All these gave the same result, showing considerable growth across the weld after prolonged annealing at  $200^{\circ}$  C., but the amount of growth observed varied very much.

Fig. 8 shows a typical example of crystals that have grown across the weld; the line of the weld is still faintly indicated by a discontinuous line, CD, probably representing an included impurity of a non-metallic character, around which the crystals have grown much as they grow around the slag in wrought-iron.

In order to remove all doubt as to the action of the impurities which were introduced, and particularly to obviate the possible contention that their action was either purely mechanical or else of the nature of that of the "dirt" more or less requisite in many chemical actions, certain further experiments on welds in lead were made. In these, the matter introduced at the weld was —

- (1) Flakes of lead.
- (2) Clean iron filings.
- (3) Clean sand.

In all three cases no growth across the weld took place, which confirms the view that the presence of a more fusible eutectic in an inter-crystalline boundary is essential to crystalline growth across that boundary. We think, therefore, that we are justified in regarding the results of these experiments as strong confirmation of the solution theory of crystalline growth in annealing.



## METALLIC ALLOYS\*

By J. E. STEAD

WHAT is a metallic alloy?

In the light of modern research, a metallic alloy is a mixture of metallic substances, which, after melting, does not separate into two separate layers.

When such a separation does occur, each layer becomes a separate and distinct alloy.

Lead and zinc, bismuth and zinc, lead and aluminium, bismuth and aluminium, cadmium and aluminium, when melted together, are instances in which separation occurs.

Some non-metallic elements, such as phosphorus and carbon, combine with metals to form definite chemical compounds possessing metallic characteristics. These take the place of metals in some alloys, and behave exactly as if they were metals.

Matthessen (*Jour. Chem. Soc.*, 1867, page 220) appears to have regarded iron and steel as alloys, and describes the carbon-iron compounds as such.

It is quite justifiable to describe phosphor-tin, phosphor-copper, phosphor ferro, spiegeleisen, cast-iron and steel as metallic alloys, for the phosphides and carbides which are present are certainly metallic substances, and alloy with the metals. It has not been the custom in the past to describe pig irons and steels as alloys, but they undoubtedly have a right to the term.

## The Constituents of Alloys

A constituent of an alloy may be defined as a separate body, which has an entity peculiarly its own.

The simple substances, brought together by fusion or other methods to form metallic alloys, do not in every case exist as elementary bodies in the cold metal. Take the case of gold and lead. When they are melted together in about equal proportions the cold alloy contains a very little, apparently, free gold; but the greater mass of it is composed of two kinds of white crystalline bodies, which are definite chemical compounds of gold and

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\* Cleveland Institution of Engineers. Lecture delivered Dec. 10, 1900.

lead, and in addition to these there is a eutectic containing, as one of its constituents, lead, in which a portion of one of the definite compounds remains in solid solution. There are very few alloys which contain in one specimen so many separate constituents. Although only the two elementary elements lead and gold are present, yet the alloy contains at least four constituents. This very remarkable alloy is briefly referred to here, for, in order to describe it, the use of practically all the general terms for the constituents of alloys has had to be employed. The constituents of alloys, therefore, in categorical order are as follows:—

1. Free metals in a pure state.
2. Solid solutions of one metal in another, or of definite chemical compounds in an excess of metal.
3. Eutectic mixtures.
4. Definite chemical compounds of metals with metals, or metals with certain non-metals.
5. Allotropic modifications of metals or of definite chemical compounds.

#### I. FREE METALS IN A PURE STATE

These are the metals which fall out of solution, or crystallize in the pure state.

They are found in the alloys as crystallites (skeleton crystals minus their crystal faces), or as perfectly formed crystals (idiomorphic) with all their true faces and angles. When present in these forms, as a rule they have been formed in a fluid matrix and were the first to fall out of solution. If the metal has a lower fusing point than that part of the alloy which first crystallized, it is found between the crystals or crystallites filling up the interstices. If the metals form a eutectic, one or all of them may be in the free state in juxtaposition.

There are two methods of ascertaining whether or not a metal, which is apparently in a pure elemental state, is really so. The first, which is the most satisfactory, is to dissolve away by some suitable solvent, everything except the apparently free metal and then make an analysis of it. Unfortunately this is not always possible, as it is difficult and often impossible to obtain a re-agent which is inactive on the metal it is sought to isolate.



The second method is a synthetical one. The metal is melted with varying minute quantities of the second metal or element, and the alloys are slowly cooled. They are then cut up, the sections are polished, and the structure developed either by polishing, "heat tinting," or etching. They are finally examined under the microscope, and if the least possible quantity of the added metal or definite compound formed by it can be detected, it may be assumed that the metal under examination does not form a solid solution with the second metal.

This method is also capable of determining exactly what amount of one metal will form a solid solution with another.

It sometimes happens that when solidification is very rapid, complete separation is not effected, even in the metal which, under normal conditions of cooling, may disentangle itself from its fellow constituents.

Many of the constituents of alloys which have hitherto been accepted as pure metals, when more perfect methods are discovered for their isolation, will probably be found to be solid solutions.

The apparently pure crystallites of silver which fall out of lead-silver alloys containing a large quantity of silver, have been proved by my friend, the late Mr. Saville Shaw, of Durham College of Science, to contain a considerable quantity of lead.

Osmond has proved that copper can retain about 1 per cent silver in solid solution, and that silver can retain the same quantity of copper.

I have elsewhere shown that iron retains phosphides of iron in solid solution, and that copper can retain a sensible amount of antimony and arsenic.

## 2. SOLID SOLUTIONS

It will have been gleaned from the previous remarks that a solid solution of one metal in another, is a solid substance containing two or more elements in such an intimate mixture that the highest power of the microscope cannot detect them on properly prepared sections.

Ostwald, in his work on solutions, defines solutions as being "homogeneous mixtures which cannot be separated by mechanical means."

It happens that many mixtures such as eutectics cannot be actually separated by mechanical means; although they can by "heat-tinting," by polishing, or by etching, be seen to be separate from each other, and if they are separate, the substance cannot be homogeneous, or be proved to contain separate independent constituents.

Many mixtures of metals which form liquid solutions do not remain in solution when they solidify. The term "solidified solution" has been applied to solid alloys of metals which were in solution when liquid, but which are not necessarily in solid solution when cold. It is important that the distinction between solidified solution and solid solution should be remembered, so as to avoid confusion.

Solid solutions crystallize in a form identical with or very closely approximating to that constituent which preponderates.

In a true liquid solution of salt in water, no microscope can detect any of the constituents: it appears to be homogeneous. Solid solutions of one metal in another are similar in that respect.

Sir William Roberts-Austen's definition of solid solution is as follows:

"A solid solution is a homogeneous mixture of two or more substances in the solid state. In metals, no one has as yet worked at non-crystalline mixtures, and solid solutions of metals when crystalline are solid 'isomorphous mixtures,' or 'mixed crystals.'"

This definition is a most concise one, and certainly appears to cover every kind of solid solution.

There are, however, several kinds of solid solutions:

- (1) In which one metal, in an alloy, on crystallizing, retains a portion of the other homogeneously diffused throughout its whole crystalline mass.
- (2) In which during crystallization the central portions of the crystals contain less of the dissolved metal than their external boundaries.
- (3) In which the metals form a definite chemical compound, a portion of which is retained in solid solution in the excess of the metal or metals.
- (4) In which the non-metallic elements form definite chemical compounds with a portion of the dissolving metal and remain in solid solution.

The terms "isomorphous" and "mixed," as applied to crys-



tals, have been and still are used synonymously, and are synonymous with the term "solid solution" in metals. The term "mixed crystals" might lead to the erroneous idea that the crystals were mixed up together, but remained independent. The term "isomorphous," according to Professor Bauerman, can only be strictly applied to mixtures of substances crystallizing in the cubic system.

There seems to be less objection to the term "solid solution." It may be applied to crystallized or non-crystallized substances, and conveys the meaning that the bodies in solution are as intimately associated as if they were in liquid solution, and that there is not complete separation of constituents when the fluid solution passes into the solid solution.

The expression "mixed crystals" should be avoided in metallurgical nomenclature if any other term is available, for the eutectics are described as eutectic mixtures, the term mixture in this case meaning an alloy or compound separated into two or more parts, whereas a so-called "mixed crystal" consists of a homogeneous whole.

As a rule, all metals and alloys are crystalline, therefore solid solutions of one or more metals in others may be accepted as crystalline solid solutions.

### 3. EUTECTICS

What is meant by the term? It was Guthrie who first applied it to a constituent of alloys.

On reading the original paper of Guthrie, it is clear that he recognized that the eutectic proportions of the metals present have not necessarily any relation to their atomic weights, and says, "of course, in many cases, metals can be fused together in simple multiples of their atomic weights, but in most such cases, one of the metals will separate out first and so destroy the ratio." . . . "When we are dealing with metals which, like antimony or arsenic, are themselves halogenous, on the one hand, or with such strongly chemico-positive metals as sodium on the other, we may and do get alloys of atomic composition or metallo-metallic salts. But the temperature of fusion of these is never, so far as I am aware, lower than that of either (both) of the constituents: they are not eutectic."

This last sentence makes it clear that one property of a eutectic, according to Guthrie, is that its melting-point must be lower than that of the mean of the separate constituents. All the instances quoted in his paper illustrate this second principle.

Recognizing that certain metals may combine to form metallo-metallic salts, Guthrie, by inductive reasoning, concluded that in an alloy such salts might be formed, and that they would take the part of a free metal and form eutectic alloys with one or other of the constituents. He says, "The very bodies resulting from the chemical union of the two metals will possibly and probably furnish starting-points of new series of eutectic alloys, consisting of a single metal on the one hand and the chemical alloy on the other."

Professor Guthrie prepared any given eutectic by melting the metals and then allowing that metal which happened to be in excess to crystallize or fall out of solution, and when the greater part had become solid, the residual alloy which still remained fluid as a kind of "mother-liquor" was poured off and allowed to set. This crude eutectic was remelted and allowed to partially solidify, and the mother-liquor was again poured off. The same treatment was repeatedly applied until the mother-liquor invariably yielded the same constant composition. This alloy when solid, but for the contradiction in terms, might have been called a solidified mother-liquor. Guthrie therefore applied the term eutectic.

A eutectic, then, must have the following properties and components:

- (1) A solidifying and melting point lower than that of the mean of its constituents.
- (2) A single melting and solidifying point.
- (3) It may consist of two or more metals which do not unite chemically, or of a metal and a definite chemical compound, and possibly of two or more definite chemical compounds (Metallo-metallic salts).

Recent research has shown that it may be constituted as follows:

- (4) It may consist of a mixture of a solid solution of one metal in another and a free metal.
- (5) It may consist of a metal solid solution of a definite chemical metallo-metallic salt, and that same metallo-metallic salt in the free state.



- (6) It may possibly consist of two solid solutions.
- (7) Is an exception to the general rule, and relates to the eutectic pearlite which is formed in solid steel when it cools slowly from  $700^{\circ}\text{C}$ . to below  $400^{\circ}\text{C}$ . It is a solid solution at  $700^{\circ}\text{C}$ ., but splits up into two constituents on cooling.
- (8) A eutectic may contain two eutectics, one formed at a solidifying point, the other of pearlite formed on cooling below  $700^{\circ}\text{C}$ . White pig iron, free from phosphorus and high in carbon, is an instance of this.

The method of Guthrie is undoubtedly the best for making the eutectic mixtures, but it necessitates using large quantities of the metals. The second in value is that in which the metal is poured into a cylindrical cast-iron mould lined with loam, and after solidification is nearly complete, it is compressed by hydraulic pressure, and the eutectic squeezed out of a suitable opening.

Sir W. Roberts-Austen has employed a similar plan to this. It is described in his "Fourth Report on Alloys" to the Institution of Mechanical Engineers, 1897.

"The alloy under examination is placed in a steel cylinder, which is fitted with two loose steel plungers, each about  $\frac{1}{2}$ -inch diameter. The whole is placed between the jaws of a hydraulic press, and a pressure of about  $\frac{1}{2}$  ton per square inch is maintained on the ends of the plungers. The cylinder is then slowly heated, the temperature at any moment being measured by a thermo-couple, which is placed in a hole drilled in the cylinder. At a certain definite temperature part of the alloy will liquefy, and can be squeezed out between the plungers and the wall of the cylinder. The temperature is noted at which the alloy is sufficiently liquid to be extruded. The heat is then raised, and a further portion of the alloy can sometimes be separated. Finally, a comparatively infusible residue is left. These several portions are then analyzed. In some cases it is found to be desirable to subject the extruded portions to a second treatment in the compressing cylinder."

It was the hydraulic method I used in expressing the liquid eutectic from Cleveland iron, described before this Institution in 1876, and it is the best in cases where a small quantity of the eutectic is in presence of a large mass of metal. A third method, useful in such cases where the fusing-point of the alloy is under  $500^{\circ}\text{C}$ ., and where only small quantities are available, has been

employed with great advantage in my laboratory in preparing the eutectics of the fusible metals.

The melted alloy is poured upon the surface of a sheet of flexible asbestos paper. When the mass has partially solidified to the consistency of a thick paste, a second sheet of asbestos is placed on the top of the metal, and a wooden ruler is rolled over the plastic mass from behind in a forward direction. The eutectic is in this way squeezed out and runs as a fluid drop in front of the ruler, leaving the solid metal behind. The crude eutectic so obtained is remelted and is again subjected to the same treatment. It is possible in this way to obtain the eutectic from 10 grammes of alloy.

The last method is that which I have used most extensively in the preparation of the eutectics containing two metals. It may be called the microscopic method. It is based upon the characteristic micro appearance of nearly all the eutectics, and on the statement of Guthrie, now generally recognized, that if one of the constituent metals is present in excess it will first solidify, leaving the residual eutectic to solidify at a lower temperature. The metal which falls out of the mother-liquor is clearly seen in the micro section when it is properly polished and etched. It generally takes some clearly marked crystalline form.

Having prepared several mixtures synthetically by melting the metals in several proportions, and allowing them to solidify slowly, pieces of each are cut, polished, etched, and examined. As a rule, it does not take more than ten minutes to prepare a specimen. A glance through the microscope reveals at once which specimen contains the greatest proportion of the eutectic and which metal is in excess. Several further alloys are then made approximating to this, but with diminishing proportions of the metal known to be in excess. The resulting mixtures are examined, and probably one of them will be found to consist of pure eutectic, free from crystals or crystallites of either of the constituent metals. A chemical analysis is then made to check the figures synthetically obtained.

It often happens that in a series of alloys of two metals, more than one eutectic is formed. A very simple method of detecting these, and also of obtaining the complete series of alloys in one single specimen, has been used with most satisfactory results in my researches.



A small special mould of fire-clay, having a cavity of 2 c.m. deep and 1 c.m. square, is heated to a temperature a little below the melting-point of the metal which is most infusible. The two metals are melted in separate crucibles, and a portion of that which has the highest gravity is poured into the mould, and when the lower part has solidified the second metal is poured in drop by drop, the following drop being added when the metal in the mould has nearly solidified or become plastic. When the mould is filled, a cold metal plate is placed on the top to hasten solidification of the last portion of the metal added. When cold, if the experiment has been conducted properly, the one metal in the pure state will be found at the bottom and the other at the top, the metal between containing a complete gradation or series of alloys. One side of the little ingot is then polished, etched, and examined. The specimen so prepared constitutes a very perfect chart or map, and is the best guide to work from in making a systematic research of the alloys of any two metals, and if more than one eutectic is present it may be recognized by its characteristic appearance.

An example of what a eutectic is (not a metallic eutectic) presents itself in common salt and water containing 23.5 per cent salt and 76.5 per cent water. This mixture when cooled to about  $22^{\circ}$  C. completely solidifies at that temperature, and a separation of salt and ice is effected simultaneously. They do not, however, separate in the sense that the salt falls to the bottom and the ice floats to the top, but separate into minute particles which then remain side by side in juxtaposition in the solid eutectic.

When snow and salt at normal temperatures are mixed together, they mutually dissolve each other, and if the proportions present are those of the eutectic, a liquid at a temperature of  $0^{\circ}$  on the Fahrenheit scale will result, and if this liquid is surrounded by a still colder liquid it will solidify and become the solid eutectic of ice and salt at  $-22^{\circ}$  C. or about  $7^{\circ}$  below zero Fahr.

Fig. 1 shows the solubility curve of salt and water.

Although cold mixtures of certain metals, lead and tin for instance, do not react upon each other at normal temperatures, if intimate mechanical mixtures are heated to the melting point of the least fusible metal, tin, the more fusible metal, lead, will dissolve in it, and the temperature will fall with the continued

solution of lead, and will not cease to melt and dissolve until the solidifying point of the eutectic is approached. The eutectics are not all of the same form; the following have been noticed:

- (1) The curviplanal, in which the constituents consist of curved plates in juxtaposition. Examples of this are to be found in the alloy of silver and copper and in slowly cooled carbon steels. Fig. 2 represents the eutectic of the latter.
- (2) The honeycombed or cellular. A very common variety, gold and lead, bismuth and tin, and many other alloys yield eutectics of this variety. Fig. 3 is that of the eutectic of phosphorus and iron.

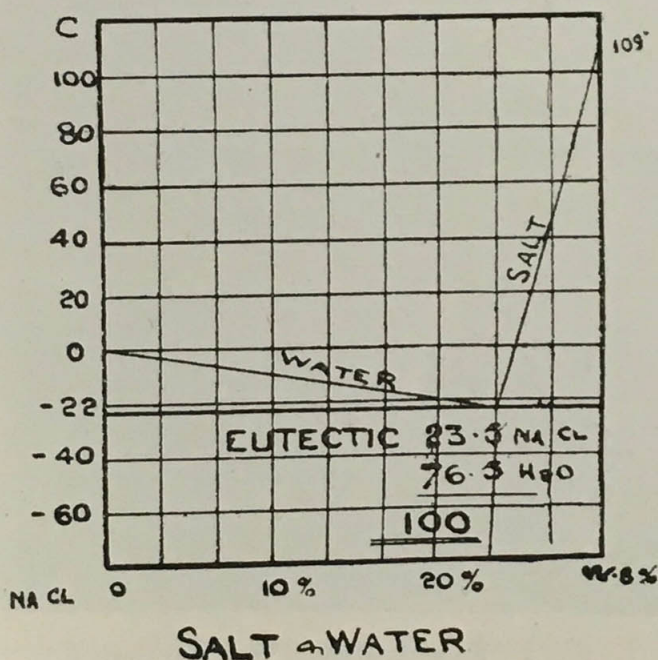


Fig. 1.

- (3) The rectiplanal, in which the two constituents separate in flat plates. Silver and lead eutectic is an example. Fig. 4 represents this.

Many of the eutectics when rapidly cooled assume a spherulitic structure. The two constituents commence to separate and solidify from nuclei and grow from these, yielding a mass resembling the spherulitic appearance of certain minerals.

Some eutectics, if allowed to cool slowly, assume geometric crystalline forms, which may be more or less completely isolated by pouring off the still fluid metal after a portion has solidified.



These may be also seen in the polished and etched sections of the solid eutectics. Lead and antimony yield spherulitic forms when rapidly cooled, and occasionally hexagonal prisms when more slowly solidified, which are built up of laminæ of lead and antimony.

The eutectic alloy of antimony, lead and tin exhibits very fine geometric forms entirely built up of separate plates of a hard

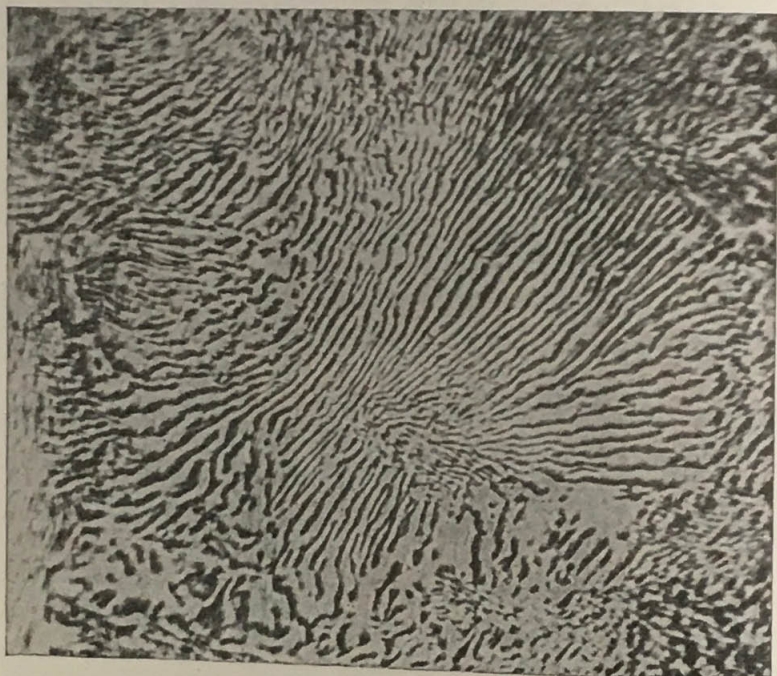


Fig. 2. Eutectic alloy of Iron and Carbon.  
Magnified 1000 diameters.

and soft constituent. The eutectic of gold and lead crystallizes in cuboidal forms if cooled slowly, but yields spherulites if rapidly solidified.

The bismuth and tin eutectic also yields crystalline forms.

It is difficult to ascertain whether the geometric forms are determined by the crystalline habit of the hard or the soft constituent. It is very probable that the former is the dominating factor, but nothing can be stated definitely until the research is more advanced.

## 4. DEFINITE CHEMICAL COMPOUNDS

Many of the metals combine chemically in atomic proportions. They also combine in the same way with certain non-metals. These are generally termed definite chemical compounds—the metallo-metallic salts of Guthrie. The eutectics are definite mechanical mixtures and consist of two or more separate constituents and are therefore different from the chemical compounds, which are quite homogeneous and are recognized by having certain characteristics different from those of the elements

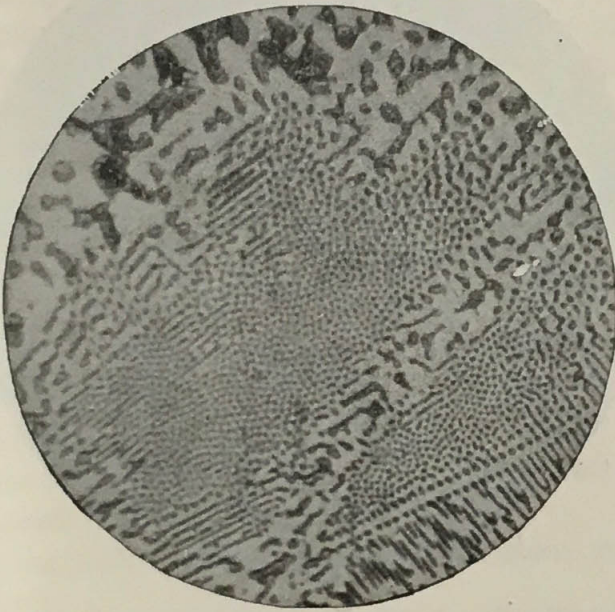


Fig. 3. Eutectic alloy of Iron and Phosphorus. Magnified 350 diameters.

alloyed, the color, hardness, toughness, brittleness, and crystalline form being often quite different. It often happens that when the metals are brought together in the molten state, great heat is evolved, an almost certain indication of chemical union. Copper and zinc, copper and aluminium, iron and phosphorus and many other couples might be advanced as instances.

Gold and aluminium yield a purple definite chemical compound, lead and gold two different chemical compounds, nearly white in color and so brittle that they can be crushed readily to powder, yet both metals in the free state are exceedingly malleable.

Copper and antimony yield two different compounds, one of a



purple color, the other a faintly yellow metal, and both compounds are very brittle.

Aluminium and copper are comparatively weak metals, one is of a red color, the other a neutral gray, yet the definite chemical compound is golden in color, is harder than either element and is much stronger and tougher. When they are brought together in the liquid state they combine with an evolution of heat.

Copper and zinc form several chemical compounds.

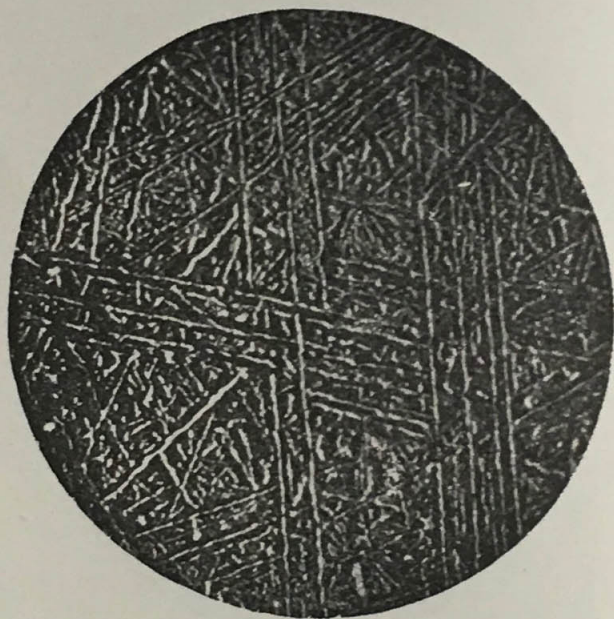


Fig. 4. Eutectic alloy of Silver and Lead. Magnified 90 diameters.

Copper and phosphorus form a pale yellow tinted compound.

Zinc, with arsenic or phosphorus, forms white compounds.

##### 5. ALLOTROPIC MODIFICATIONS OF METALS OR OF DEFINITE CHEMICAL COMPOUNDS

Bricks of the same kind in the hands of a builder may be fashioned into many varied structures; in like manner nature can and does group or build up the same kind of atoms to form molecular structures widely different from each other.

When such variations in a simple substance occur, the different forms are called allotropic modifications. Carbon atoms grouped differently yield the diamond, graphite and charcoal. Sulphur yields a yellow and brittle variety and a plastic brown modification. Phosphorus yellow and red allotropic modifications.

Many of the pure metals can be obtained in two or more states. Iron at different temperatures can be obtained in three allotropic states; one at normal temperature, a second at about  $780^{\circ}$ , and a third at a temperature above  $870^{\circ}$  C. Tin, gold and many other metals may exist in more than one modification. Matthessen believed that one or both the metals in binary alloys were allotropically different from the metals before alloying them, but it is by no means very easy to determine whether or not such is really the case.

Modern research has shown that some of the metals chemically combine with each other and that they sometimes form solid solutions.

This would lead one to hesitate in accepting Matthessen's conclusions, for, if chemical action intervenes, the atoms of the metals must be reformed into new groupings and such reorganization may be sufficient to alter the properties of the metals independently of the allotropic change.

We have also yet to learn much about the effect of one metal being dissolved in solid solution in another.

We do not really know in what way the atoms are grouped in solid solutions.

The peculiar way in which some of them behave towards chemical solvents, might lead to the conclusion that the dissolved metals or metallic substances were included in the molecular groupings.

For instance when phosphide of iron in iron is in a free state capable of being detected by the microscope, it is left insoluble on dissolving the alloy in dilute acid, but if it is in solid solution in the iron, in the same acid a non-metallic residue is left insoluble, containing much more phosphorous than the phosphide, and appears to be a complicated decomposition product, in fact, formed from a compound molecular group of phosphide of iron and iron atoms and the dilute acid.

In annealed carbon steels, dilute acid likewise behaves in a



similar manner, and leaves the carbide of iron insoluble; but if the same steel is heated to bright redness and quenched, the acid leaves a hydrated carbon residue free from carbide. It is believed that the hardened steel contains the carbide in solid solutions.

It is quite possible, however, in both the cases referred to, that the carbide and phosphide in solid solution may be in such an attenuated state that the acid, incapable of acting upon the more massive particles, does act upon the finely divided liberated definite compounds, and that the acid test is not a proof that they are included in the molecular groupings with the solvent iron.

It is also more than probable that, when in solid solution, the definite compounds are allotropically altered, and that the modified molecules have the property of being decomposed by dilute acid.

These reflections show what a large field is open in this branch of metallurgy for patient original research and what need there is for more knowledge before definite conclusions can be formed.

It has been argued by some authorities that, in certain cases, because one of the metals, liberated from solid alloys by solvents or other means, is undoubtedly in an allotropic state different from the normal, it is evidence that they existed as such in the alloys.

If the metals were in chemical union in the alloys with each other and acid removed one of the metals, the others which were left would most probably be in an allotropic state, probably in monatomic molecules, or at any rate, in more simple groups than they were in the normal metals; but that would be no evidence that they were allotropic in the alloys, for the allotropic forms might depend for their existence on the agency employed to decompose the compound chemical molecules.

The alloy of iron and nickel containing 25 per cent nickel, according to Dr. J. S. Hopkinson (*Roy. Soc.*, Vol. XLVIII. 1890, p. 23), when heated to  $500^{\circ}\text{C}$ . and cooled either rapidly or slowly has no magnetic properties, but if cooled below  $0^{\circ}\text{C}$ . it can be attracted by a magnet. The passage from the non-magnetic state is accompanied by an increase of hardness, a decrease of electrical resistance, and a decrease of density from 8.15 to 7.98. Mr. Osmond has since confirmed these results.

Osmond has shown that when a steel with about 1.5 per cent carbon is quenched in ice water, it is composed of two constituents, martensite and austenite. When this metal is immersed for a few minutes in liquid air, its properties are materially altered. Its density is lowered from 7.798 to 7.692 — or 1000 cubic inches expand to about 1014 cubic inches. In addition to this its magnetic permeability and residual magnetism are increased.

These transformations which can scarcely be attributed to chemical change can only be explained by the assumption that there has been a regrouping of the atoms. They are most marked instances of allotropic change in the alloys.

The hardness of hardened steel has been attributed to the same change brought about by the influence of dissolved carbide in solid solution. There seems good reason to believe that such is the case, considering that, although the amount of the dissolved hard carbide in the hardened carbon steel is under 15 per cent, the steel is actually as hard, and, according to some authorities, harder than the carbide. That the iron in such material is allotropically changed, and that the intense hardness is a property of the modified state of the iron atoms, appears to be a reasonable conclusion, but there is still wanting experimental evidence to place it beyond all doubt: indeed, we know very little of the molecular constitution of metals and alloys, but as there is more and more interest being taken by the most highly trained scientific workers, we can look forward with confidence to the future, knowing that in time, the obscure will be made clear.

### **Methods Employed for Studying the Constitution and Properties of Alloys**

In modern research, there are several methods in use for studying the changes which take place when an alloy passes from the liquid to the solid state, and for ascertaining the constitution and properties of the alloy when cold. A few of these are as follows:

1. Thermal method.
2. Microscopic method.
3. Chemical methods, analytical and synthetical.
4. Mechanical methods.
5. Electric and magnetic methods.



## I. THERMAL METHOD OF EXAMINING ALLOYS

The method used by Sir W. Roberts-Austen for making autographic records of cooling alloys, etc., may be briefly described as follows:

A Le Chatelier platinum and platinum-rhodium couple, properly protected by a sheath of fire-clay, having been inserted in the slowly cooling metal, the thermo-electric current produced is conveyed by wires to a D'Arsonval dead-beat galvanometer, on the mirror of which a ray of light is caused to fall. This ray is reflected in a camera upon a photographically sensitized glass plate, which is caused to travel either upwards or downwards by clock-work or by a water float. The ray of light moves horizontally, the plate vertically.

When the alloy has passed through all of its thermal changes and the plate fallen out of range of the ray of light, it is removed and the latent record is developed by suitable developing solutions.

The movement of the plate is always maintained at a regular rate, and the speed is carefully noted. The vertical record is ruled off by lines at distances corresponding to seconds or minutes. The value of the vertical distances in equivalent temperatures having been previously determined, lines cutting the horizontal time ordinates at right angles are also drawn. From this chart containing, as coördinates, time and temperature, the thermal changes can be read.

A primary cooling curve may be described as the resultant of two movements at right angles to each other.

If these are equal, the resultant will be a straight line at an angle of  $45^\circ$  to the direction of motion.

If the temperature movement is irregular, the resultant will be a curve or several curves, or curves and straight lines. If the temperature movement is arrested altogether, a straight vertical line will be made as long as the arrest continues.

The record, whether it consists of a straight line or a curve or a combination of straight lines and curves, for convenience is termed a curve.

If it has been obtained when the substance under examination is cooling, it is termed a cooling curve; if when it was heating up, a heating curve.

It is usual in reproducing the records to turn them at right angles to the direction they were taken, so that the vertical divisions give the time and the horizontal the temperature.

When a record of a red hot body, like copper, cooling in the air, is taken, in consequence of the greater difference between its temperature and that of its environment when red hot, compared with the same metal when it is approaching the temperature of

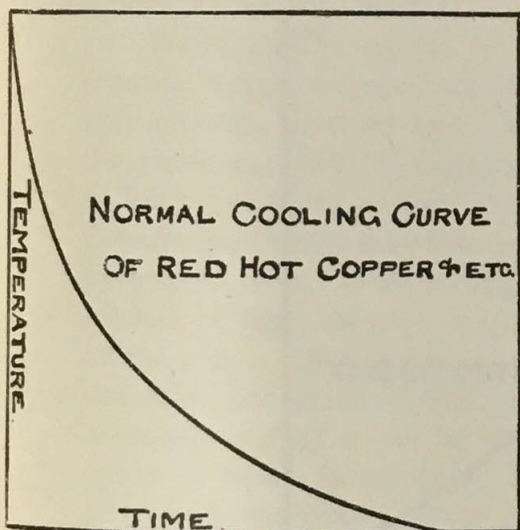


Fig. 5.

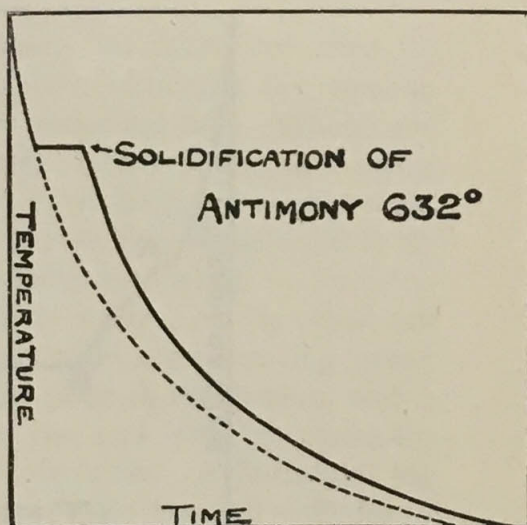


Fig. 6.

the surrounding air, the cooling curve is steep at first and gradually becomes less so, and more nearly approaches a horizontal position as it cools, as shown in Fig. 5.

If pure liquid antimony at  $800^{\circ}$  C. is tested by this method, the curve will commence by making a rapid descent at first until the temperature falls to the solidifying point of antimony ( $632^{\circ}$  C.), but as soon as it is reached, the metal begins to solidify and in doing so gives out its latent heat of fusion, and, until it is all quite solid, it remains at that temperature, and the curve at the change turns abruptly and travels in a horizontal direction.



When the metal is quite solid, it then takes the same course as it does when the red-hot copper cools. (See Fig. 6.)

When an alloy of equal parts of lead and antimony is allowed to solidify the cooling curve is very different. Antimony in the proportion of about 13 per cent and lead 87 per cent is a eutectic mixture, and this solidifies at  $247^{\circ}$  C. Pure lead has a melting point of  $325^{\circ}$  C. Antimony one of  $632^{\circ}$  C. The mixture of equal parts of each metal contains 57.5 per cent of the eutectic and 42.5 per cent antimony. In cooling, this 42.5 per cent antimony will solidify completely before the 57.5 per cent

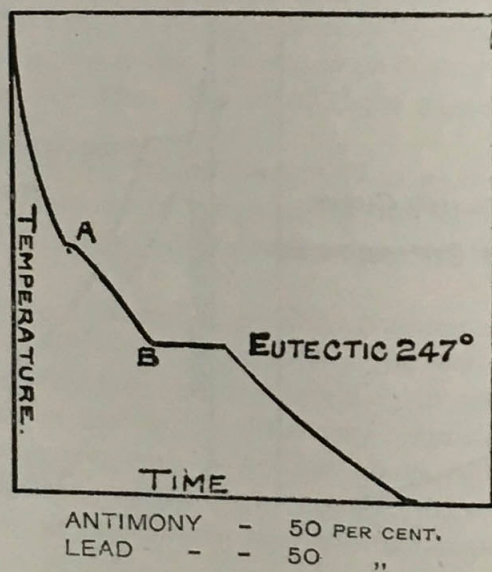


Fig. 7.

eutectic can begin to freeze, but its solvent power for antimony prevents any of that metal from falling out of solution till the temperature is far below the melting point of antimony. The point at which it does begin to fall out of solution is approximately when the mean of the melting points of the eutectic and metal is reached. This being so, the first portion of excess of antimony in our 50 per cent alloy should fall out of solution at the mean of the melting points of 42.5 per cent antimony and 57.5 per cent of the eutectic, or about  $409^{\circ}$  C.

The curve yielded on the recording pyrometer is shown in Fig. 7.

It will be noticed that the first deviation, from the type of the symmetrical normal curve of cooling solid copper is not abrupt, but is rounded at the top, and, after the first arrest, the curve does not return to the normal track as it does after a pure metal completely solidifies. This is easily explained. The first part only of the excess of metal antimony falls out of solution at A, the remainder solidifies continuously between the points A and B, and it is not till the temperature falls to B that the whole of it is out of solution. Heat, therefore, must be evolved over the period represented by the distance between A and B, hence the peculiarity of the curve.

When all the excess of antimony has fallen out, then the eutectic begins to freeze and during its solidification the temperature remains constant and yields a horizontal line. When solid the curve assumes its normal course. I have arranged a model of Sir W. Roberts-Austen's recording pyrometer on a large scale. It is on the platform before you. On one side of the room is the pyrometer with the reflecting galvanometer, which is throwing a beam of light on a screen, 42 inches square, on the other side of the room. The screen is covered with white drawing paper, and takes the place of the sensitized plate in the camera, and is arranged so that it can be made to rise and fall by a clock, the speed of which is regulated by an air brake. A horizontal bar is fixed between the screen guides. A pen charged with ink is fixed in a saddle, and this can be moved across the face of the screen on the horizontal bar. When the screen is stationary and the saddle is moved across its face, a plain horizontal line is marked upon it. When the saddle is still, and the screen moves, a vertical line is the result. At this moment the spot or band of light rests stationary on the right hand of the screen. It represents the temperature of molten lead at about  $400^{\circ}$  C., for the thermo couple is placed in a crucible containing lead maintained at that temperature by a gas jet placed below it. I now turn out the gas so as to allow the lead to cool and solidify, and at the same time start the clock. You will see at once that the spot of light is moving to the left, and as it is incapable of tracing its own track, I draw the saddle with the ink style attached along the horizontal bar, always keeping the pen on the spot of light.



As both the screen and the light are moving, the ink makes a slightly curved line, diagonal in direction. Now the spot of light has ceased to move the lead is beginning to solidify. It remains stationary for about thirty seconds, and then begins to move, indicating that the whole of the lead has become solid, and the movement continues until the light has passed off the sheet altogether. The ink record, you will see, clearly indicates the great thermal arrest. The curve traced resembles that shown in diagram No. 3.

It is quite immaterial whether the track of the curve is from the right or the left. It is, however, generally drawn from the left hand side falling towards the right, as is shown.

The complete equilibrium curve showing the arrests of a series of two or more metals is constructed by plotting the arrests on a chart in which composition and temperature are coördinates, the vertical lines representing temperatures, the horizontal spaces composition. The base horizontal line is divided into equal spaces corresponding to the proportion of metals present; the lines, vertical to the base, are divided into equal spaces of temperature. Having obtained a complete set of time-temperature records of many different mixtures of the two metals, ranging from 100 per cent of one metal to 100 per cent of the other, the temperature arrests are marked off on vertical lines drawn from the points on the base line corresponding to the composition of the alloys. Lines are then drawn through the several records corresponding to the points of first arrest, and a second line is drawn through the points of second arrest. If there is a series of third arrests, a line is drawn through these also. The chart is in this way completed.

Take the case of copper and silver, a carefully determined curve by Messrs. Heycock and Neville, Sydney College, Cambridge. Mixtures of the two metals gave the following arrests:

Silver per cent	Copper per cent	First freezing point °C.	Second freezing point °C.
0.00	100.00	1081.5	
12.99	87.01	1019.6	
18.39	81.61	996.5	
28.60	71.40	953.5	
37.64	62.36	917.4	
42.03	57.97	900.2	
58.51	41.49	837.2	778.4
61.71	38.29	823.7	778.6
71.90	28.10	778.6	778.2
76.4	23.6	797	778.6
82.1	17.9	825	
85.9	14.1	847	
89.5	10.5	870	
96.48	3.52	926.1	
100.00	0.00	960.0	

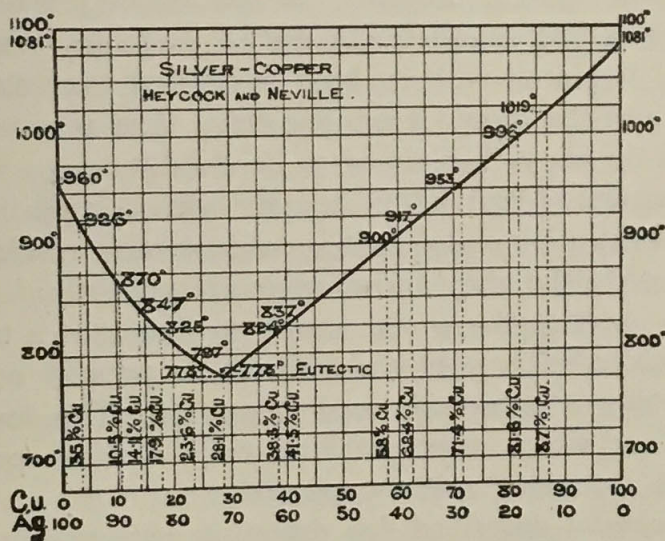


Fig. 8.

The curve constructed from these figures is given in Fig. 8.

The curve method also throws light upon the allotropic changes, which occur when the change of state is accompanied by an absorption or evolution of heat.

In the proceedings of this institution in February, 1900, I explained the methods of polishing and microscopically examining metals; it is not necessary to repeat them now, and therefore I refer you to that paper.

There are, however, some points of interest in micrographic



technology, which should be referred to here. The first is a method of preparing suitable bright and smooth surfaces of the more fusible metals without the necessity of polishing them. This consists in casting them on glass or polished steel, a method used by Prof. Ewing and Mr. Rosenhain, and described by them in a paper on the crystalline structure of metals before the Royal Society. *Philosophical Transactions*, Series A, Vol. 193, page 354.\*

The smooth polished surface of glass or steel leaves a similar surface on the metals cast on them.

It is important that the glass should be heated to approximately the same temperature as the molten metal poured upon it, otherwise it will fly to pieces.

More recently, Mr. H. J. Hanover, of Copenhagen, has described in the *Bulletin de la Société d'Encouragement pour l'Industrie Nationale*, 1900, 6, 210-211,† a method of preparing similar surfaces by casting on sheets of mica. A conical shaped mould of charcoal is placed with its base on heated mica and the alloy is poured into the mould from the top. The result in the case of the lead-tin alloys is claimed to be good.

For antimony-lead alloys the process is modified. After having cast the alloy on mica, it is covered with cyanide of potassium, and when the alloy and cyanide are quite fluid, a condition obtained by heating the surface with the flame of a blow-pipe, a second piece of mica, previously heated, is pressed on the liquid metal and allowed to remain until it has set. This leaves a good upper surface which after washing may, without polishing, be etched and examined microscopically.

The method described for detecting eutectics in alloys has long been used in my laboratory with highly satisfactory results. It is one which must take a primary position in metallographic work. Its value has been independently discovered by Professor H. Le Chatelier, who has described it in the *Bulletin de la Société d'Encouragement pour l'Industrie Nationale*, Sept. 30, 1900.‡

The system of superimposing two liquid metals to determine the rate of diffusion of one metal into the other is now well known

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\* *The Metallographist*, Vol. III, page 94.

† *The Metallographist*, Vol. IV, page 29.

‡ *The Metallographist*, Vol. IV, page 1.

by the publication of Sir W. Roberts-Austen's classical researches before the Royal Society, but the micro-examination of the complete graded series of solid alloys made by the method, for the object of detecting the various constituents, had not, so far as I know, been referred to in any lecture or public work previous to 1898, when I explained it to the Newcastle Section of the Society of Chemical Industry, and gave preliminary examples of its value.

It was also described in my paper on Iron and Phosphorus, read before the Iron and Steel Institute at Paris, September 18, 1900.\*

Very many of the alloys which contain a fusible metal, although they can be obtained in a complete series by the superimposing method, require different treatment in different parts to develop the structure: for instance, the alloys of copper-tin and copper-zinc, two of the most important series of any, contain copper with small quantity of zinc or tin at one end and fusible metals at the other, and the structure of the high copper alloys are best developed by heating so as to yield oxidation tints, but heating the complete series causes the zinc or tin to melt and extrude from the polished surfaces. It is necessary in such cases to make two or more series commencing with copper at one end and the alloy with about 40 per cent copper at the other, a second with about 40 per cent copper at one end and about 25 per cent at the other, and so on. In this way, several gradated series are formed, each set of which can have its structure developed by the same treatment.

In this way, in the zinc-copper series, which I have long been studying, the structures in the sections change more gradually, and it is easier to study them than when all are contracted into one piece.

The relation between a short and extended spectrum in spectroscopic work, and a short and extended gradation sample in the alloys, is strictly comparable.

After examining microscopically any series, the object which should always be aimed at is to correlate structure and composition.

Each specimen is marked at various points, photographs

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\* *The Metallographist*, Vol. IV, page 89.



taken, and by means of a hard hack saw the section is sawn at the points marked, and the metallic sawdust is carefully analyzed after removing any broken saw teeth, which may be present, with a magnet.

Having thus obtained structure and composition, a series of corresponding alloys may be made synthetically and the thermal and other properties determined.

Mr. Charpy and Professor H. Le Chatelier have recently stated that alumina prepared by calcining ammonia alum is a first-rate material for polishing metals. The diamondine powder made in Switzerland, and used by many jewellers for polishing steel, consists of pure alumina and is what I have used since 1894, and all the sets of apparatus, which have been supplied by Messrs. Carling for polishing metals, includes a small bottle of it. It is satisfactory to have the independent testimony of two high French authorities of the value of the substance I have recommended for years.

If one part of a micro-section wears away more rapidly in polishing than another, that alone is proof of the existence of at least two parts of varying softness. When one constituent or more is left in relief by either polishing or etching, and the light is caused to fall on the surface, at an angle with it, the sides or edges of the parts in relief reflect the light up the microscope tube, and *they* only appear to be illuminated.

If there is no relief or irregularity of the surface, none of the incident rays are reflected into the microscope, and the object then appears to be perfectly dark.

If one part of an object darkens, or is more rapidly etched or corroded, excepting in certain cases where electric action occurs between the constituents, or assumes color tints on being heated in air or another gas, more rapidly than other parts, it is proof of a variation in the constitution of the alloy.

When a section treated as just described, shows a *gradation* in the coloring or corrosion in any part of it, it is an almost certain proof of the presence of a solid solution of one constituent in another, in which the solution is not equable throughout.

Certain alloys of silver and copper, antimony and copper, copper and tin, etc., are notable instances of such irregular solid solutions.

When eutectics are present in large proportion in alloys, on

polishing with or without etching, as a rule with unaided vision, they appear to be beautifully colored, and resemble mother of pearl or opal.

When such opalescent tints are noticed, it may be taken as strong evidence, but not absolute proof, of the presence of a eutectic. In some cases, the laminæ, of which most of the crystalline metals are built, on etching strongly, sometimes are acted upon irregularly, the alternate edges being left more or less in relief. This gives rise to an interference of light, and gives a chromatic appearance.

Homogeneous metals, however, on polishing by Osmond's method, never yield iridescent tints, whilst almost always, if not invariably, eutectics do so.

Many metallographists ascertain the relative hardness of the various constituents revealed by the microscope by the simple process of drawing the point of a hard sewing needle across the surface of the section, and then examining the scratch. Where the indication or furrow is deepest, that part may be regarded as the softest; where the needle has not sunk so deep, it is considered to be harder.

### 3. THE CHEMICAL METHOD

(a) *The Analytical Method.* — In many cases, it is possible, as has been before shown, to separate the constituents present by chemical solvents, and, having obtained them in an isolated state, to make chemical analyses. In this way their composition is determined.

Dilute acetic acid has been used by Mr. Saville Shaw and myself for separating the free lead from alloys of that metal and silver, by Mr. Shaw, and from gold in my researches. The action is extremely slow, and must be continued for weeks, and sometimes for months, before the lead is perfectly removed.

Dilute nitric acid is useful in some cases. Concentrated nitric acid does not act upon one of the definite compounds of lead and gold, which is by this means isolated.

Nitro-hydrochloric acid rapidly dissolves free tri-ferric phosphide, but has less action on the di-ferric phosphide. They can be separated by means of that reagent.

Hydrochloric acid diluted with water removes the free tin



from tin-antimony alloys leaving insoluble the tin-antimony compounds. It also removes tin from the alloys of tin and phosphorus, and tin and arsenic, leaving the definite chemical metallic compounds insoluble.

Cyanide of potassium dissolves the free gold from lead-gold alloys rich in gold.

Many more instances might be given, but the above will be sufficient to show generally the system of analytical separation of the constituents.

(b) *The Synthetical Method.* — Definite compounds, such as the purple compound of aluminium and gold, discovered by Sir W. Roberts-Austen, and some of the copper-aluminium compounds and many other definite compounds, which do not suffer decomposition when they are melted, may have their composition determined synthetically with the assistance of the microscope. This is effected by melting the metals in definite proportions, and noting whether the structure of the mixture at any given point or points is homogeneous. When homogeneous compounds are produced and the metals are present in simple atomic proportions, it is generally assumed that they are definite chemical compounds. It is possible, however, that there are exceptions to this rule, for it may be that in some cases solid solutions of metals and metals, or metals and definite compounds, may contain the metals in atomic proportions, yet not be chemical compounds.

#### 4. MECHANICAL COMPOUNDS

The elasticity, tenacity, and ductility of alloys and resistance to crushing strains in conjunction with other correlated properties are useful in assisting to a correct conclusion regarding the constitution of alloys.

As the methods of testing are so well known to engineers, it is not necessary to refer to them.

#### 5. ELECTRICAL AND MAGNETIC METHODS

The difference in electrical conductivity and the magnetic properties of some alloys, before and after variations in heat treatment, lead to the conclusion that such treatment has influenced a reorganization of the molecular structure, with the production

of allotropic modifications. Slight traces of one metal alloyed with another generally alters the electric conductivity, and when present in large quantities profoundly affect its electrical properties.

Students should study the works of Matthessen, M. Le Chatelier, Osmond, Burrett, and others, that they may understand the value of electrical and magnetic methods of testing alloys.

### Classification of Binary Alloys

Mr. H. Le Chatelier (*The Metallographist*, Vol. I, No. 2, pages 94-5) has grouped the various binary alloys according to the character of their curves of fusibility into three main classes.

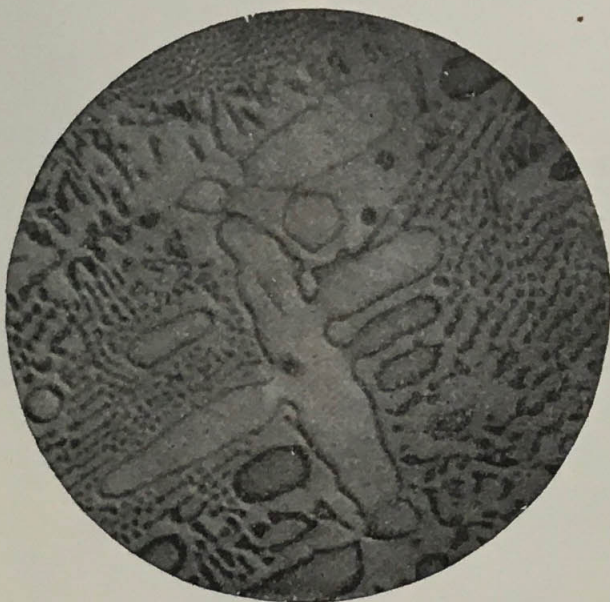


Fig. 9. Ingot metal containing about 8 per cent phosphorus. The broad light parts are crystallites of the metal containing about 1.7 per cent phosphorus. The white mottled ground mass is the eutectic containing 10.2 per cent phosphorus. Magnified 250 diameters.

(1) Two branches of curves starting from the melting points of the pure metals and meeting at a point corresponding to the eutectic alloy; this curve is obtained when the two metals form neither definite combinations nor isomorphous mixtures. Examples are yielded by alloys of antimony and lead, copper and silver.



In the alloys of this class, one metal falls out of solution on the first; the other on the second branch; the eutectic solidifies where the two branches meet.

The photos of the alloys of iron and phosphorus, copper and antimony, show the structures of the solid alloys at the points where the two branches meet and on each side of the junction.

Fig. 3 is the eutectic of iron and phosphorus. On the iron

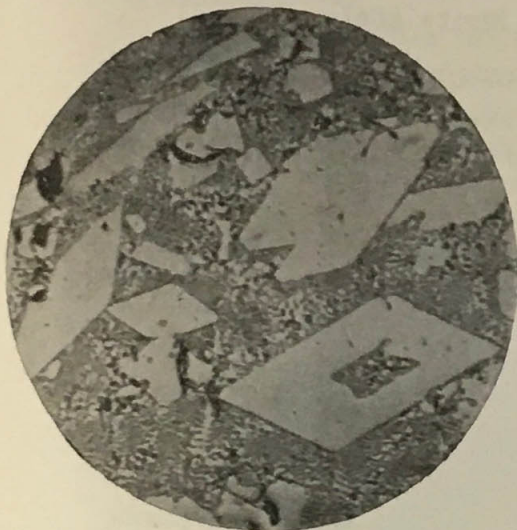


Fig. 10. Ingot containing 11.10 per cent phosphorus and 88.9 per cent iron, showing crystals of  $\text{Fe}_3\text{P}$  embedded in a ground mass of the eutectic. Magnified 60 diameters.

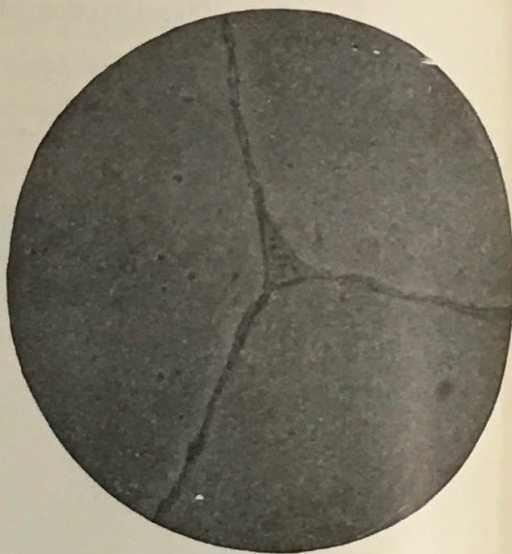


Fig. 11. Ingot metal with 1.8 per cent phosphorus, showing portions of three contiguous grains surrounded by a cellular envelope of  $\text{Fe}_3\text{P}$  with a little of the eutectic at the part where the three grains meet. Magnified 350 diameters.

branch, crystallites of iron, containing a little phosphide of iron in solid solution, embedded in the eutectic will be noticed in Fig. 9. On the phosphide of iron branch, crystals of phosphide of iron appear also embedded in the eutectic (Fig. 10). Fig. 11 represents grains of the solid solution surrounded with the eutectic.

The freezing or cooling points of iron and phosphorus containing between 0 per cent and 16 per cent of phosphorus, although they have not yet been taken in a systematic manner, resemble those of copper, silver, antimony and lead, etc.

As the phosphorus is increased, the first freezing point is steadily lowered until the eutectic point is reached; afterward the point of solidification is raised, till the compound is composed entirely of phosphide of iron  $\text{Fe}_2\text{P}$ .

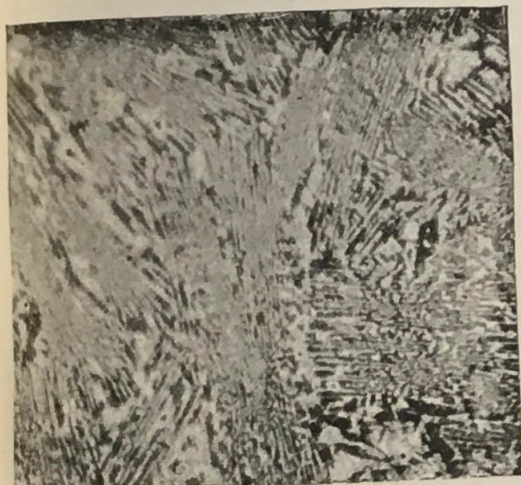


Fig. 12. Eutectic alloy of copper 24.2 per cent and antimony 75.8 per cent. Magnified. 100 diam.

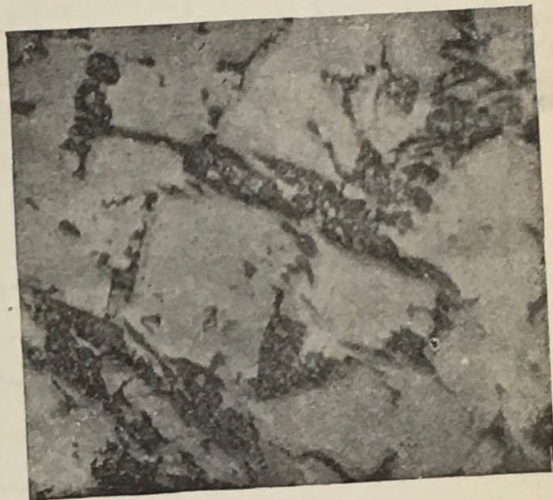


Fig. 13. Copper 10 per cent, antimony 90 per cent. Magnified 100 diameters.



Fig. 14. Copper 20 per cent, antimony 80 per cent. Magnified 100 diameters.

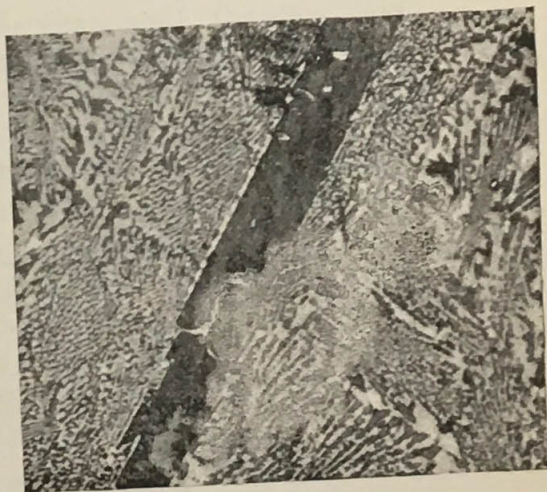


Fig. 15. Copper 30 per cent, antimony 70 per cent. Magnified 100 diameters.

The alloys of phosphide of iron and iron up to that point are strictly comparable with the binary alloys of the first class of Chatelier.

Figs. 12 to 15 illustrate the eutectic and the alloys on each



side of it, one containing antimony, and the other the purple definite compound of antimony and copper, both embedded in the eutectic.

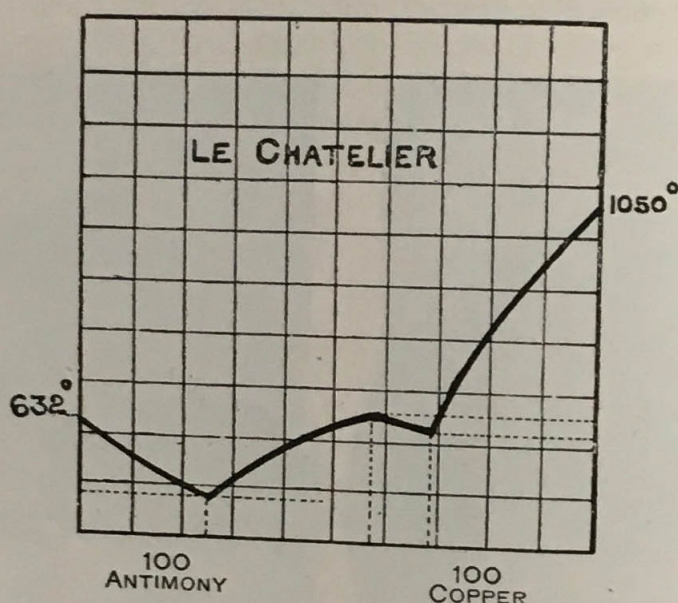


Fig. 16.

In this series the alloys between 0 per cent and 51.5° of copper are typical in the way they solidify, and of their micro-structure of binary alloys of the first class, but the whole series from 0 per cent to 100 per cent copper really belongs to the second class of Chatelier.

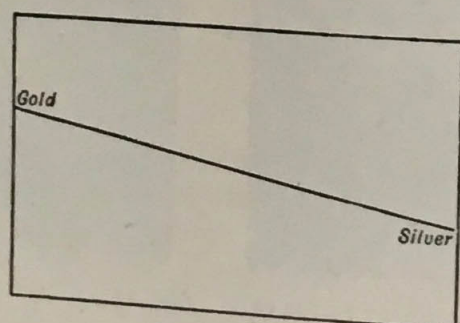


Fig. 17.

The white crystals in Figs. 13 and 14 are antimony, the dark band in Fig. 15 is a crystallite of the definite compound  $\text{SbCu}_2$ . Fig. 12 is the eutectic with 24.2 per cent Sb.

(2) Three branches of curves, two of them starting from the melting points of the pure metals, and a third exhibiting a maximum, and crossing the former two in two points corresponding to two eutectic alloys; it is the case of two metals forming a definite combination. Examples are given by the alloys, gold and aluminium, copper and antimony. (Fig. 16.)

(3) The curve of fusibility is continuous, and unites the melting points of the two metals; it is the case of two metals forming isomorphous mixtures. Examples are yielded by alloys of gold and silver, and antimony and bismuth. (Fig. 17.)

Although the approximate cooling curves of the alloys of classes 2 and 3 are given, it is not intended to describe them further in this lecture.

It is highly probable that as knowledge is extended, the curve method of research will enable many more classes to be introduced. Although much has been learned in recent years, there is yet much more to learn before a systematic classification can be safely attempted.

### Alloy Charts for Representing the Structural, Constitutional and Physical Properties of Alloys

Fig. 18 is a chart from which the constitutional composition, the melting points, and the micro-structure of all the lead-antimony alloys can be seen at a glance. The upper part of the diagram shows the proportion of the antimony and eutectic on one side, and the lead and eutectic on the other. Composition and percentage are coördinates. This system, diagrammatically showing the constitution of alloys, I have long used in my lectures. (It has been independently recommended by Mr. Albert Sauveur of Boston, editor of *The Metallographist*.)

For instance, if it is desired to know the constitution, etc., of say, an alloy of 50 per cent lead and 50 per cent antimony, trace a vertical line through the chart from the horizontal composition line where it is marked 50 per cent lead. Where this line cuts the cooling curve, draw a line at right angles to it, till it joins the temperature scale. The degrees of heat at which the alloys begin to solidify will thus be found. Where the vertical line cuts the triangle on the upper chart, draw also a line at right angles,



till it joins the percentage columns. The proportion of eutectic and antimony will be given on the scales, the former at the right, the latter on the left.

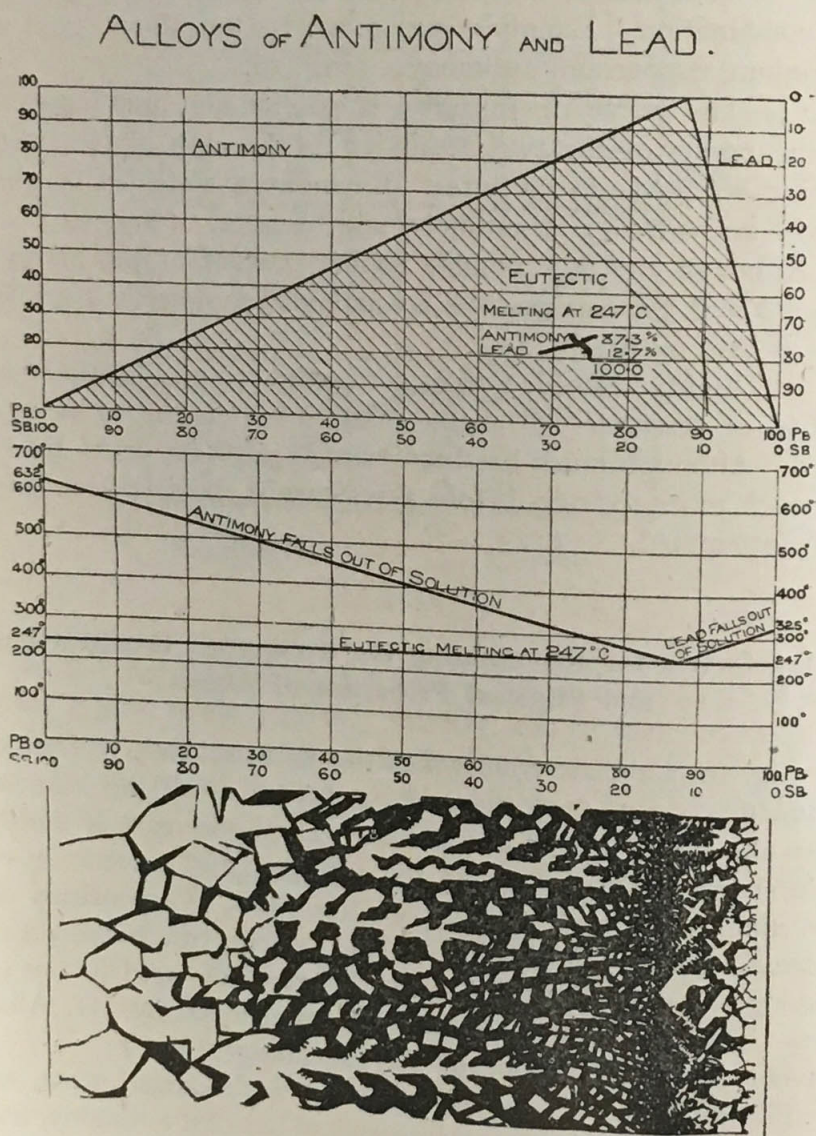


Fig. 18.

Where the vertical line cuts the lower diagram, represents approximately the microstructure of the 50 per cent by 50 per cent alloy. The particulars are as follows:

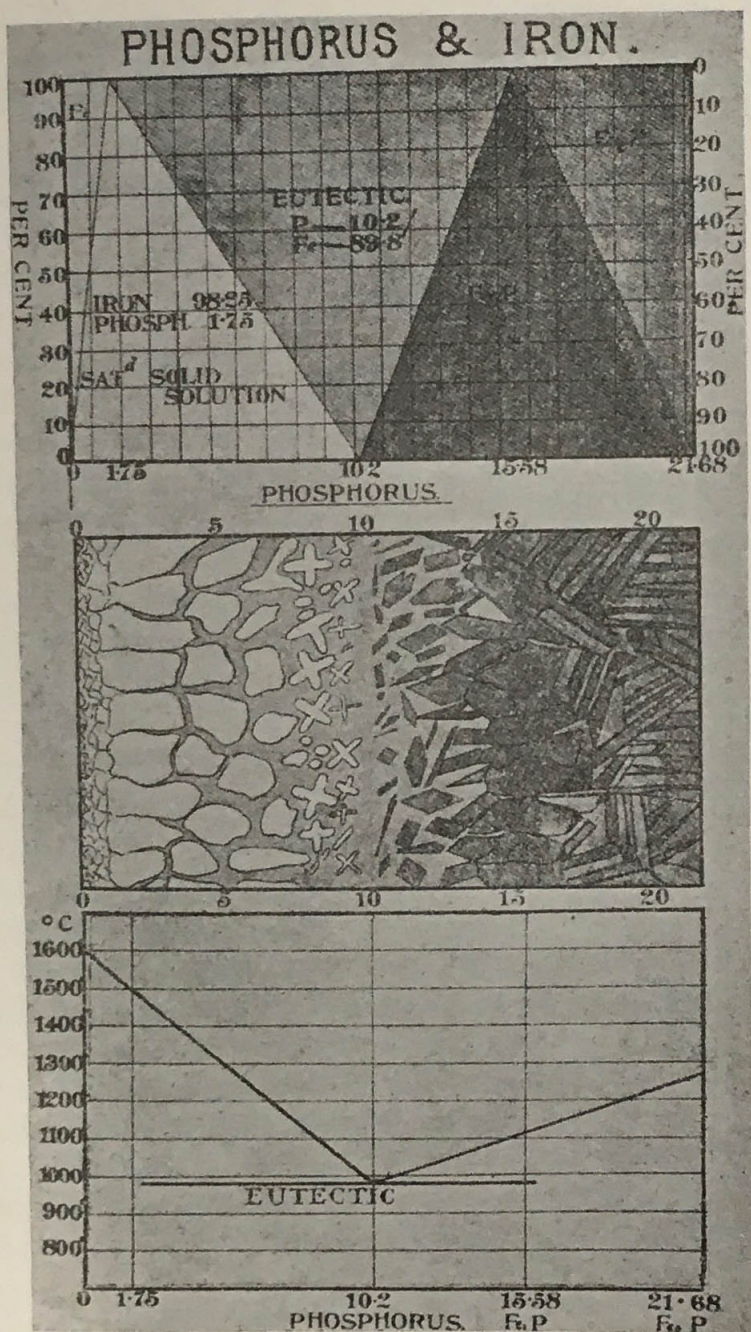


Fig. 19.

Eutectic .....	57.5
Metallic antimony crystals.....	42.5
	<hr/>
	100.0
First freezing point.....	409°
Second (or eutectic) freezing point.....	247°



The chart should also have another diagram showing the mechanical properties, but as I have not got the data, I cannot append it.

If future metallurgical works on alloys were to be provided with charts similar to what have been described, they would greatly simplify the work of the student.

Fig. 19 gives similar information regarding some of the iron and phosphorous metallic alloys.

The cooling curve in this case is approximate, and is only meant to indicate generally that the melting point falls with each addition of phosphorus till the eutectic point is reached about  $980^{\circ}$  C., and then rises as the phosphorus is further increased.

Arrangements are in course for the accurate determination of the various melting points.

Much of the data of this lecture is the result of the researches of Mr. Osmond, Mr. Le Chatelier, Mr. Charpy, Sir W. Roberts-Austen, Messrs. Heycock and Neville, Dr. Stansfield, Mr. Saville Shaw, and others.

To the student of metallurgy I would give the advice that the published writings of these gentlemen should be carefully perused.

The only periodical published which is confined exclusively to this branch of modern research is *The Metallographist*, edited by Albert Sauveur, of Boston, U. S. A., and if the original papers of the gentlemen above named are not available and beyond reach, that periodical should be obtained, for it gives excellent abstracts and reviews of the most recent metallographic researches.

The only book published in the English language, so far as I know, which contains descriptions of the methods of modern research in alloys is that of Sir W. Roberts-Austen, viz., "Introduction to the Study of Metallurgy," a work which should be in the hands of every metallurgical student.

## THE NOMENCLATURE OF METALLOGRAPHY\*

IN view of the fact that, with the development of metallography, the nomenclature is becoming more and more involved, the Council of the Iron and Steel Institute, at the instigation of Mr. J. E. Stead, appointed a Committee consisting of Mr. William Whitwell, President (chairman), Mr. F. W. Harbord (Englefield Green), Mr. E. Heyn (Charlottenburg), Mr. T. W. Hogg (Newburn), Professor H. M. Howe (New York), Baron H. von Jüptner (Donawitz, Austria), Professor H. Le Chatelier (Paris), Professor A. Martens (Charlottenburg), Mr. Walter Rosenhain (Birmingham), Professor Albert Sauveur (Boston), Mr. E. H. Saniter (Middlesbrough), Dr. A. Stansfield (Montreal), Mr. J. E. Stead (Middlesbrough), and Mr. Bennett H. Brough, Secretary, to consider the matter and to ascertain whether it would be possible to take steps to make the terminology less complicated and more precise.

The Committee has not met yet,<sup>†</sup> but it has been decided to draw up a glossary, in the hope that it will tend to promote the unification of terms, the simplification of those used, and the elimination of many of them. It is hoped, too, that the glossary may be improved, before final publication in the *Journal of the Iron and Steel Institute*, by suggestions from members interested in the matter. Such suggestions, whether additional terms or better definitions, are earnestly invited by the Committee.

As far as possible, the exact equivalents in French and German will be added. This addition will, it is hoped, prove of great value to those who are in the habit of consulting Continental memoirs in the original. It will at the same time be of assistance to the editor of the great International Technical Lexicon, now being prepared under the direction and at the cost of the Society of German Engineers, a society which, with its roll of 16,000 members, is the largest engineering society in the world. The Iron and Steel Institute has undertaken to coöperate as far as possible in this great work, and it is thought that in drawing

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\* *Iron and Steel Institute*, September, 1901.

† Since this was written the Committee has held two meetings.



up an authoritative glossary of the most recent branch of the metallurgy of iron, the Iron and Steel Institute will be rendering valuable aid.

Based upon the microscopic examination of thin sections of minerals and rocks, observations were recorded in 1858 by Dr. H. C. Sorby, member of the Iron and Steel Institute, in a paper on the microscopic structure of crystals, indicating the origin of minerals and rocks (*Quarterly Journal of the Geological Society*, Vol. XIV, page 453), and in October, 1867, by the late Mr. David Forbes, member of Council and Foreign Secretary of the Iron and Steel Institute. These observations gave birth to the special science of petrography. In view of the fact that metallic bodies are analogous to rocks, the exact knowledge of metals called for the creation of a corresponding science of metallography, in which the pioneers were Dr. Sorby, whose publications go back to 1864, and Professor Martens, whose publications go back to 1878. In 1880 the use of the microscope was introduced at the Le Creusot works, and the investigations of Mr. F. Osmond and Mr. J. Werth were started, and have been continued since that time along the path indicated by Dr. Sorby. Metallography is cultivated to-day in the principal metallurgical countries. Starting from the scientific laboratory, it has been extended further and further into works laboratories, where it will undoubtedly become an indispensable auxiliary to chemical analysis and physical tests.

In view of its close analogy to petrography and to the study of meteoric irons, metallography necessitates the use of similar technical terms, and consequently wherever possible the terms familiar to the mineralogist and geologist should be used in describing the structures of metals and alloys, and the coining of new words is to be deprecated.

The following provisional alphabetical list, which contains the more important terms used by authors of memoirs dealing with metallography, has been prepared for the consideration of the Committee, and is now issued in the hope of obtaining criticisms and suggestions from the members of the institute, in order that the Committee may have before them data upon which to base their judgment.

## Glossary of Terms

Ac<sub>1</sub>, Ac<sub>2</sub>, Ac<sub>3</sub>, etc. Critical points. Points of flexure in the heating curves of iron and steel.

ACICULAR. — (Ger. *Nadlig*, Fr. *Aciculaire*.)

A term applied to needle crystals found in drusy cavities of ferro-manganese, basic slags, etc. It has been applied erroneously to describe the appearance of sections of plates and wedge-shaped crystallites in certain metals and alloys. This and similar terms of Latin or Greek derivation might perhaps advantageously be replaced by their equivalents of Saxon origin.

AIR PITS. — (Ger. *Luftgrübchen*.)

An epithet applied to microscopic air bubbles, which have taken a geometrical form, found on the surface of certain metals cast on glass (Ewing and Rosenhain). This term might be discarded.

ALLOTRIOMORPHIC. — (Ger. *Allotriomorph*, Fr. *Allotriomorphe*.)

A term applied to crystals which have taken their shape from their surroundings.

ALLOTROPIC FORM OR MODIFICATION. — (Ger. *Allotrope Form*, Fr. *Forme allotropique*.)

A term applied to an element which has physical properties and chemical activities different to the normal state of that element.

ALLOTROPY. — (Ger. *Allotropie*, Fr. *Allotropie* — Faraday.)

Is the capacity to undergo without change of composition a change of chemical and physical properties.

"Allotropy is a change of internal energy occurring in an element at a critical temperature unaccompanied by a change of state" (Roberts-Austen).

ALLOY, METALLIC. — (Ger. *Legierung*, Fr. *Alliage métallique*.)

An intimate mixture or union of metallic substances which on melting do not separate into two distinct liquid layers (Stead).

ALPHA IRON. — (Ger. *Alpha-Eisen*, Fr. *Fer alpha* — Osmond.)

The normal condition of pure iron below the temperature of 750° C. Crystallizes in the cubic system. (See Beta and Gamma iron.)

AMORPHOUS. — (Ger. *Amorph*, Fr. *Amorphe*.)

Non-crystallized. Not formed by crystallization. A term that should never be applied to alloys; no alloy can be said to be amorphous.

APHANITIC. — (Ger. *Aphanitisch*, Fr. *Aphanitique*.)

A term applied by geologists to a texture in which the component grains are so minute as to become visible only under the microscope. This term might be applied to the finest structure of hardened steel instead of the word "amorphous." It is, however, of questionable utility.



Ar<sub>1</sub>, Ar<sub>2</sub>, Ar<sub>3</sub>, etc. Critical points. Points of flexure in the cooling curves of iron and steel.

ARBORESCENT.—(Ger. *Tannenbaumförmig*, Fr. *Arborescent*.)

Tree-like; a term applied to certain crystallites with the appearance of fir trees, and which sometimes occur in drusy cavities in metals or embedded in solid alloys.

ATOMIC VOLUME.—(Ger. *Atomvolumen*, Fr. *Volume atomique*.)

The atomic volume of an element is its atomic weight divided by its specific gravity.

AUSTENITE.—(Ger. *Austenit*, Fr. *Austenite*—Osmond.)

A constituent of steel softer and less magnetic than martensite, with which it is often associated. It is produced by quenching small sections of steel containing more than 1.5 per cent carbon in ice-cold water from a temperature of 1100° C.

The structure of such quenched steel is developed by polishing on parchment moistened with liquorice solution or nitrate of ammonia. The austenite remains white, the martensite becomes brown, and appears in a zigzag form. A sewing-needle drawn across the section scratches the austenite more deeply than the martensite.

On account of its low degree of hardness, and because it appears to be stable only at the highest temperatures, Baron Jüptner is led to consider it as a solution of elementary carbon, or of ionized carbide of iron in iron.

BACILLAR.—(Ger. *Stäbchenförmig*, Fr. *Bacillaire*.)

Rod-shaped. A term that might be discarded.

BETA IRON.—(Ger. *Beta-Eisen*, Fr. *Fer bêta*—Osmond.)

An allotropic non-magnetic condition of pure iron, existing at temperatures between 750° and 860° C. It is isomorphous with alpha iron, crystallizing in the cubic system. When beta iron changes to alpha iron heat is evolved and magnetic properties are developed.

BLOWHOLES.—(Ger. *Blasenhohlräume*, *Blasen*, Fr. *Soufflures*.)

Small cavities, spherical or ellipsoidal, found in ingots of cast metals. They consist of bubbles of gas which have not been able to escape before the metal became completely solid.

BRITTLENESS.—(Ger. *Brüchigkeit*, Fr. *Fragilité*.)

More or less easily broken. It is caused by want of cohesion between the cleavage planes of the crystalline grains or between their joints. It may be produced in tough ductile metals by the presence of a brittle foreign substance which envelops the grains. The track of fracture in such case generally follows the brittle envelopes.

BURNED.—(Ger. *Verbrannt*, Fr. *Brulé*.)

This term is applied to metal which is brittle in consequence of a chemical alteration of its mass caused by excessive heating.

Recent study has shown that a so-called burnt metal such as steel or iron is not necessarily oxidized in its mass. It is specifically lighter than the same metal previous to overheating, owing to

the evolution of gases in the solid metal which causes an incipient separation of the crystal grains. Near to the external surface, if carbon is very low or absent, intergranular layers of oxide of iron may sometimes be detected. The chemical composition is not necessarily altered by the so-called burning. Steel may, on heating to a very high temperature in an inert atmosphere, develop many of the properties of burned steel after cooling. In such cases oxidation, of course, is impossible. Burned steel is generally coarsely granular, and is easily fractured.

**CANCELLED.** — (Ger. *Gitterförmig*, Fr. *Formé en treillis*.)

Latticed; crossed by two series of parallel lines—a structure sometimes developed on etching alloys. This term might be discarded.

**CARBON.** — (Ger. *Kohlenstoff*, Fr. *Carbone*.)

To this most important element occurring in iron and steel several names have been given.

**CARBIDE CARBON.** — (Ger. *Carbidkohle*, Fr. *Carbone du carbure* — Ledebur.)

The condition in which the carbon exists in cementite, synonymous with combined carbon.

**CARBON ANNEALING.** — (Ger. *Temperkohle*, Fr. *Carbone de recuit* — Osmond and Werth.)

The finely divided carbon having graphite properties, which separates from white cast iron and from certain steels during prolonged annealing, and according to Ledebur, from high carbon steel when it is raised to a red heat by rapid hammering. It is insoluble in dilute acids. Ledebur describes this carbon as temper carbon, the word "temper" having in Germany a different meaning to what it has in England. Annealing carbon is the term that best expresses Ledebur's meaning.

**GRAPHITE CARBON.** — (Ger. *Graphit*, Fr. *Graphite*.)

The carbon which separates from highly carburized fluid irons, previous to solidification and at the solidifying point.

**MISSING CARBON.** — (Hogg.)

That part of the carbon in hardened and tempered steels which does not give color on dissolving the steel in nitric acid 1.20 sp. gr.

**HARDENING CARBON.** — (Ger. *Härtungskohlenstoff*, Fr. *Carbone de trempe*.)

The condition of the carbon when it confers hardness to steel, as in steels chilled from above  $A_{r1}$ . It is, like carbide carbon, combined carbon. Baron Jüptner distinguishes three varieties of combined carbon: bi-, tri-, and tetra-carbon.

**CELLULAR.** — (Ger. *Zellig*, Fr. *Cellulaire*.)

Containing irregular spheroidal or ellipsoidal cavities. Applied by Osmond to net-like structure.

**CEMENTITE.** — (Ger. *Cementit*, Fr. *Cémentite*.)

This term was first applied by Professor Howe to carbide of iron in the separate state as a constituent of steel. The researches of



Abel, Müller, Osmond, and others show it to have the formula  $\text{Fe}_3\text{C}$ . The term may be, however, and actually has been, used to embrace all the separate carbides in cast iron and steel containing manganese, chromium, etc. The free carbides in nearly all commercial metals and steels, as a rule, are not composed of  $\text{Fe}_3\text{C}$  alone, but are admixtures in molecular union with  $\text{Mn}_3\text{C}$ . Cementite may exist in fine granules, thin plates, or in comparatively large masses in steel and pig metal.

According to Osmond, it is the hardest constituent (No. 6 Mohs scale) in metal and steel, not colorable by polishing and etching with dilute nitric acid, iodine solution, hydrochloric acid in alcohol, etc.

Sauveur distinguishes "segregated" and "free" cementite; the former is a constituent of pearlite, while the latter occurs independently. This distinction was noticed by Sorby. As the term "segregated" suggests separation, and as one authority has called free cementite "segregated," it appears advisable to discard the word segregated and substitute the term "pearlite-cementite."

CLEAVAGE. — (Ger. *Spaltbarkeit*, Fr. *Clivage*.)

As applied to minerals and metals, is the property possessed by crystals and crystal grains of separating along certain planes. These cleavage planes are usually parallel to one or more of the faces of the crystal forms, but have not necessarily any relation to the faces of the crystal grains.

COHESION. — (Ger. *Kohäsion*, Fr. *Cohésion*.)

One of the various forces of attraction; that force by which particles are held together.

CONCHOIDAL. — (Ger. *Muschlig*, Fr. *Conchoïdal*.)

Having the shape of a shell: applied to the concave and convex fractures of some alloys of zinc-copper and tin-copper, glassy slags, etc.

CONGEALED SOLUTION. — (Ger. *Erstarrte Lösung*, Fr. *Solution congelée*.)

Synonym with "solidified solution." A homogeneous solution after solidification, irrespective as to whether the constituents of the solution in the latter state form a mixture or a solid solution in the true sense of the term.

CONSTITUENTS. — (Ger. *Bestandtheile*, *Gefügebestandtheile*, *Gefügebildner*, Fr. *Constituants*.)

As applied to metallography, are the structural parts of which alloys and metallic substances are built. Alloys containing only two or more elements or components may contain three or more constituents.

COOLING CURVES. — (Ger. *Abkühlungskurve*, *Kühlungskurve*, Fr. *Courbe de refroidissement*.)

A diagrammatic representation of thermal changes which occur when liquid or solid substances cool from a higher to a lower temperature, and in which time and temperature are coördinates.

Cooling curves may be represented in several ways: (1) By taking as coördinates the temperature "T" and the time "t" from the commencement of cooling: the curve so obtained will then more or less approximate to a parabolic curve. (2) By taking as coördinates the temperature of the metal "T," and the space of time necessary for cooling down from that temperature through a definite number of degrees: this is expressed by  $\frac{dt}{dT}$ . (3) One can choose for coördinates the temperature and the difference between the time required for the cooling of the metal under investigation, and the time necessary for some other metal (*e.g.* platinum) to cool. These latter might be termed differential cooling curves (Ger. *Differentialabkühlungskurven.*)

CRITICAL POINTS. — (Ger. *Kritische Punkte, Haltepunkte*, Fr. *Points critiques.*)

The point or zone at which a physical or chemical change takes place, as when in cooling pure iron and steel from 900° C., an evolution of heat, indicative of a physical change, occurs at the critical points Ar<sub>3</sub> and Ar<sub>2</sub>. When the change is not at one fixed point, but extends over several degrees, the area of change is called a "zone."

CRYSTAL. — (Ger. *Krystall*, Fr. *Cristal*.)

According to Professor H. Bauerman, the leading property of crystals, as distinguished from mere geometrical solids, is the invariability of the angles between corresponding faces in different individuals of the same substance.

Mr. Stead points out that the term has been used by some authorities for particles of crystals, allotriomorphic crystals, pseudomorphic crystals and crystallites. It is very desirable that the term should be confined to true idiomorphic crystals only.

CRYSTAL GRAIN. — (Ger. *Krystallkorn*, Fr. *Grain cristallin.*)

An allotriomorphic crystal, or a fragment of a crystal devoid of its crystal faces and angles. This term is to be preferred to "crystalline grain."

CRYSTALLINE. — (Ger. *Krystallin*, Fr. *Cristallin.*)

This term is used by crystallographers to denote the physical properties of crystallised matter, and is applied to any body, or portion of a body, possessing those properties without regard to its form. By petrographers, on the other hand, the same word is used to denote mineral aggregates which consist of crystallised substances, whether in the form of perfect crystals or merely as grains possessing the physical properties of crystals.

CRYSTALLISED. — (Ger. *Krystallisirt*, Fr. *Cristallisé.*)

This term is applied to that which has been produced by a process of crystallisation, no matter what the external form may be, having the physical properties of crystallised matter, applicable to any body or portion of a body possessing those properties without regard to its external form.



CRYSTALLITE.—(Ger. *Krystallit*, Fr. *Cristallite*.)

This term, first proposed by Sir James Hall, has been adopted by modern petrographers with slightly varying meanings. By some it is used as a general designation for all the indefinitely crystalline or incipient forms of individualisation of minerals. In metallography it may be defined as an imperfect crystal, in which the plane faces and angles are not developed.

CUBOIDAL.—(Ger. *Würfelförmig*, Fr. *à peu près de la forme d'un cube*.)

Applied to crystals which appear to be cubical in certain micro-sections of alloys; only applicable tentatively until the crystals are measured. Mr. Rosenhain regards this as an objectionable coined word.

CUNEIFORM or CUNEATE.—(Ger. *Keilförmig*, Fr. *Cunéiforme*.)

Wedge-shaped applicable to crystallites in many alloys prepared by the superposition method. This term might be discarded.

CUSPIDATE.—(Ger. *Speerförmig*, Fr. *Terminé en pointe*.)

Spear-shaped; tapering abruptly. This term might be discarded.

DENDRITIC.—(Ger. *Dendritisch*, *Verästelt*, Fr. *Dendritique*.)

Tree-like; a term applied to certain crystallite forms common in many metals and alloys. (See "Arborescent.")

DRUSE.—(Ger. *Druse*, Fr. *Géode*.)

A term used in descriptive metallography for cavities into which freely terminated crystals or crystallites project; common in metals and alloys.

DUCTILITY.—(Ger. *Dehnbarkeit*, Fr. *Ductilité*.)

The property of metals to elongate and bend. Ductile metals and alloys have been described as those in which sliding can take place along cleavage planes without separation occurring.

ELASTIC LIMIT.—(Ger. *Elasticitätsgrenze*, Fr. *Limite d'élasticité*.)

Is the maximum stress a substance will bear without suffering permanent distortion or fracture. This is expressed in tons per square inch or in kilogrammes per square millimetre.

ELASTICITY.—(Ger. *Elasticität*, Fr. *Elasticité*.)

According to Stewart and Gee, is that property in virtue of which a solid body tends to recover its size and shape, and a fluid body its size, after having been submitted to the action of a disturbing force.

ELASTICITY, MODULES OF.—(Ger. *Elasticitätsmodul*, Fr. *Coefficient d'élasticité*.)

Young's modulus may be described (Stewart and Gee) as follows:

If a rod or wire of length  $L$  be stretched until it becomes of length  $L + l$ , then  $\frac{l}{L}$  is the extension per unit of length. If the force causing extension be  $P$  units, and the wire or rod have a sectional area containing  $a$  units, then the force or stress is  $\frac{P}{a}$  per unit of cross-section, and

$$\text{Young's modulus} = \frac{\text{stress}}{\text{strain}} = \frac{\frac{P}{a}}{\frac{l}{L}} = \frac{PL}{aL} = M.$$

In iron and steel the value of Young's modulus is about 13,000 tons per square inch; in other words a stress of one ton per square inch produces an extension which is  $\frac{1}{1300}$  of the original length.

#### EMBRITTLING.

A term applied by Professor Howe to such elements as produce brittleness when added to steel.

EQUILIBRIUM CURVE.—(Ger. *Gleichgewichtskurve*, Fr. *Courbe d'équilibre*.)

This is a curve showing the relation of temperature and composition of a heterogeneous system, in which the phases present are in equilibrium with each other. An *approximation* to such a curve can be obtained by plotting a series of cooling curves, but in order to obtain a true equilibrium curve in this way, it would be necessary for the cooling to be infinitely slow.

The expression "equilibrium curves" probably dates from some time back, and might therefore be found in the works of Gibbs, Van t'Hoff, Van der Laar, Ostwald, Le Chatelier, etc. In any case, this term is used to denote other curves, *e.g.*, temperature-pressure-curves in the case of equilibrium between ice, water, and steam.

ETCHING.—(Ger. *Aetzen*, Fr. *Attaque chimique*.)

In order to develop the constitutional and crystalline structure of metals and alloys, the polished surfaces are subjected to action by suitable reagents, such as nitric acid, iodine, sulphuric and hydrochloric acids, etc. This action is termed etching.

ETCHING FIGURES.—(Ger. *Aetzfiguren*, Fr. *Contours d'attaque*.)

According to Baumhauer, figures, generally hollowed out pits, which are obtained by the suitable etching of crystalline surfaces, of polished surfaces, or of the surfaces of cleft crystals. They have the very closest connection with the orientation of the respective surfaces of the crystal. On crystallographically equivalent surfaces the figures, both with regard to their form as well as to the orientation, are uniform, but upon surfaces which differ crystallographically the figures differ. They are an unmistakable means for deciphering the crystalline structure of the body in the case of "crystallized bodies." In amorphous bodies they are not met with. Their size varies (and their form, too, in some cases) with the duration of the etching process, and according to the etching medium employed, for which reason it is thought that they cannot in general be identical with the crystal elements which build up the crystal.

EUTECTIC.—(Ger. *Eutektisch*, Fr. *Eutectique*.)

A term used by Guthrie (1875) to indicate solidified mother-



liquor. All eutectics have a freezing-point below that of the mean of their constituents. The German word can only be used as an adjective; the English word is often used as a noun.

**EUTECTIC ALLOY.**—(Ger. *Eutektische Legirung*, Fr. *Alliage eutectique*.)

A term defined by Guthrie (*Philosophical Magazine*, June, 1884). Stead describes three different structures in eutectics—the “curved plates,” “flat plates,” and cellular or honeycombed. As a rule, eutectic alloys, after polishing and etching, have, without the aid of a microscope, a mother-of-pearl or opal appearance when obliquely illuminated. Many eutectics in solidifying take true crystalline forms.

**EUTECTIC MIXTURE.**—(Ger. *Eutektische Mischung*, Fr. *Mélange eutectique*.)

According to Baron Jüptner, a eutectic after solidification and separation into its constituent parts, which remain in micro-juxtaposition.

**EUTECTIC POINT.**—(Ger. *Eutektischer Punkt*, Fr. *Pointe eutectique*.)

The common point of intersection of two inclined branches, and an approximately horizontal line in the freezing-point curve. This horizontal line is called “eutectic line.” (Ger. *Eutektische Linie*, Fr. *Ligne eutectique*.) The alloy, the composition of which corresponds with the eutectic point, is called *eutectic alloy*; the temperature corresponding with this point in the freezing-point curve is termed *eutectic temperature*.

**EUTECTIC SOLUTIONS.**—(Ger. *Eutektische Lösungen*, Fr. *Solutions eutectiques*—Jüptner.)

Eutectics previous to solidification.

**FERRITE.**—(Ger. *Ferrit*, Fr. *Ferrite*.)

A term first used by Howe for pure iron. It is now commonly used for that part of steel or iron containing no carbide, or at least not more than traces in solid solution. It covers, therefore, iron which may or may not contain silicon, manganese, nickel, etc., which form solid solutions or isomorphous crystallized mixtures with iron.

Ferrite is the softest structure-constituent. The unmistakable characteristic is the occurrence of cuboidal etching figures subsequent to treatment with nitric acid or copper-ammonium chloride (1 to 12).

**FIBROUS.**—(Ger. *Fasrig*, Schnig, Fr. *Fibreux*.)

Composed of fibres or threads. Fractured surfaces of some wrought irons and steels and other metals may falsely indicate fibrous structures. In such cases the fibrous fractured surface is the result of tension which draws out the crystal grains into threads or fibres.

**FISSILE.**—(Ger. *Blättrig spaltbar*, Fr. *Clivable*.)

Admitting of being split into laminae.

**FOLIATED.**—(Ger. *Blättrig*, Fr. *Lamellaire*.)

Composed of thin layers or leaves.

FRACTURE.—(Ger. *Bruch*, Fr. *Cassure*.)

The broken surface of a metal or alloy. It may be either of the following:

CONCHOIDAL.—(Ger. *Muschlig*, Fr. *Conchoïdale*.)

HACKLY.—(Ger. *Hackig*, Fr. *Hachée*.)

ROUGH.—(Ger. *Rauh*, Fr. *Inégale*.)

SMOOTH.—(Ger. *Eben*, Fr. *Unie*.)

SPLINTERY.—(Ger. *Splittrig*, Fr. *Ecailleuse*.)

FREEZING-POINT CURVE.—(Ger. *Erstarrungspunktskurve*, *Gefrierpunktskurve*, Fr. *Courbe de fusibilité*.)

A diagrammatic representation showing the summary of a complete set of cooling curves of a series of alloys consisting of two or more metals or metallic substances, temperature and composition being coördinates. (See "Equilibrium Curve.")

FRIABLE.—(Ger. *Bröcklig*, Fr. *Friable*.)

Easily crumbled; reducible to powder.

GAMMA IRON.—(Ger. *Gamma-Eisen*, Fr. *Fer gamma*—Osmond.)

An allotropic non-magnetic condition of pure iron existing at temperatures above 850° C. It crystallizes in the cubic system and its crystalline forms are combinations of the cube and octahedron (but more frequently of the octahedron) derived from the cube. In passing from the state of gamma iron to beta iron an evolution of heat occurs.

GLASS-HARDNESS.—(Ger. *Glasharte*, Fr. *Dureté du verre*.)

The utmost degree of hardness which steel is capable of receiving on quenching from above A<sub>1</sub>.

GLIDING-PLANE.—(Ger. *Gleitfläche*, Fr. *Plan de clivage*.)

A definite direction in a crystal in which the molecules glide over one another when it is subjected to a suitably directed pressure. The subject was investigated by Ewing and Rosenhain (*Proc. Royal Society*, March 16, 1899). A sharp distinction is drawn between "deformation occurring with twin formations" and "deformation by translation" (Ger.). If a change of form has occurred due to twin formation, this can be traced by means of etching figures subsequent to polishing the material. This cannot be done, however, in the case of a change of form by "slip." The change is then only noticeable if the deformation occurs subsequent to the polishing. It is rendered apparent by the slip-bands. These disappear immediately on etching, and no further trace is to be seen. The etching figures are not altered in appearance from the former ones.

GRAIN.—(Ger. *Korn*, Fr. *Grain*.)

A grain may or may not be crystalline; it may be a rounded or irregularly shaped particle, either separate or joined to other grains. Metallurgists commonly use the term, with qualifications, to describe the fractures of steel, such as fine grain, coarse grain, etc., etc. As all such steels are crystalline, the term "grain" when applied to metals may be considered as a contraction for crystalline grain or crystal grain.



GRANULAR. — (Ger. *Körnig*, Fr. *Grenu*.)

Composed of grains of irregular crystalline particles, as sandstone, marble, granite, iron, and steel, etc.

GRANULITIC. — (Ger. *Granulitisch*, Fr. *Granulitique*.)

Michel-Lévy applies this term to holocrystalline rocks composed of juxtaposed grains, all approximately of the same size and independently oriented. Iron and steel, many metals and alloys, exhibit similar characteristics.

GRINDING. — (Ger. *Schleifen*, Fr. *Adoucir*.)

The process of preparing smooth surfaces by abrasion.

GROUND-MASS. — (Ger. *Grundmasse*, Fr. *Masse fondamentale*.)

The ground-mass of a rock is the matrix in which crystals are embedded. Similarly in metallography, the term may be used for the mass which is in preponderating quantity.

HACKLY. — (Ger. *Hackig*, Fr. *Haché*.)

Rough; having fine short and sharp points on the surface; as a hackly fracture, e.g., the fracture of the native metals. Annealed steels give a hackly fracture. Coarse grained.

HARDENING. — (Ger. *Härten*, Fr. *Trempé*.)

The process of heating steel to above  $A_{r1}$  and suddenly quenching in water.

HARDENITE. — (Ger. *Hardenit*, Fr. *Hardenite*.)

According to Howe, this is understood to be martensite in the form of the eutectic solution. The term was first applied in 1897 by Charpy to martensite with its maximum percentage of carbon (0.89 per cent.). The term should be discarded.

HARDNESS. — (Ger. *Härte*, Fr. *Dureté*.)

By hardness is understood the resistance offered by a body to the separation of its particles. The hardness of a mineral is measured by the force required to scratch it with a steel point or other sharp pointed fragment of some harder mineral. The results obtained from one and the same mineral are found to vary slightly with the crystalline face experimented on, and even with the direction on one and the same face.

Relative hardness may be expressed by reference to the following scale (Mohs') :

- |                |                |
|----------------|----------------|
| 1. Talc.       | 6. Orthoclase. |
| 2. Rock Salt.  | 7. Quartz.     |
| 3. Calcite.    | 8. Topaz.      |
| 4. Fluor Spar. | 9. Corundum.   |
| 5. Apatite.    | 10. Diamond.   |

Behrens uses sharply pointed needles of different hardness to determine relative hardness, the materials employed having the following values on Mohs' scale Lead, 1; tin, 1.7; tin containing iron, 2; hard lead, 1.5 to 2.2; zinc, 2.5; copper, 3; brass wire, 3.1; gun-metal, 3.3; bronze with 12 per cent. of tin, 3.5; bronze with 18 per cent. of tin, 3.7; iron wire, 3.7 to 3.9; sewing needles, 5 to 5.5; needles tempered yellow, 4; tempered blue, 5; drill steel

tempered yellow, 6; chrome steel, 6.2 to 6.5; and ferro-chrome, 7 to 7.3. Brinell's method of determining hardness is fully described in Mr. Wahlberg's papers read before the Iron and Steel Institute. (See "Natural Hardness," "Glass Hardness," and "Tempered Hardness.")

HEATING CURVE. — (Ger. *Erhitzungskurve*, Fr. *Courbe d'échauffement*.)

A diagrammatic representation of thermal changes which occur when solid substances are heated, and in which time and temperature are coördinates. (See "Cooling Curve" and "Equilibrium Curve.")

HEAT-TINTING. — (Ger. *Anlassen*, Fr. *Oxydation par chauffage*.)

The process of heating polished surfaces of metals and alloys in air or other gas until the constituents become differently colored.

HEAT-TINTS. — (Ger. *Anlauffarben*, Fr. *Colorations de recuit*.)

The tints produced by heat-tinting.

HOLO-CRYSTALLINE. — (Ger. *Vollkrystallin*, Fr. *Holocristallin*.)

Composed wholly of crystalline material, having no interstitial matter. Pure iron, copper, and other useful solid metals, and practically all solid alloys, are holo-crystalline.

HONEYCOMBED. — (Ger. *Blasig*, *Bienenwabig*, *Wabig*, Fr. *Venteux*.)

Unsound from many blowholes. A term applied to ingots and castings.

HYPER-EUTECTIC STEEL. — (Osmond.)

Steel containing more than the eutectic proportion of 0.90 per cent. carbon.

HYPO-EUTECTIC STEEL. — (Osmond.)

Steel containing less than the eutectic proportion of 0.90 per cent. carbon.

HYSTERESIS. — (Ger. *Hysteresis*, Fr. *Hystérésis* — Ewing.)

Retardation of magnetisation (magnetic hysteresis). The persistency with which certain bodies tend to retain a previous condition. Professor Ewing uses the word in the more general sense of a dissipation of energy occurring in any cycle of operations.

IDIOMORPHIC. — (Ger. *Idiomorph*, Fr. *Idiomorphe*.)

Crystals which have developed their external forms freely.

IMBRICATED. — (Ger. *Dachziegelartig*, Fr. *Imbriqué*.)

Overlapping like the slates of a roof. Such structures are often developed on the surfaces of polished metals and alloys by etching. This term might be discarded.

INCLUSIONS. — (Ger. *Einschlüsse*, Fr. *Inclusions*.)

Many metals contain inclusions of gas and mechanically suspended foreign matter.

INTERCRYSTALLINE. — (Ger. *Zwischen Krystallen liegend oder verlaufend*, Fr. *Inter cristallin*.)

Between crystals. Applied to the eutectic and other substances in certain alloys surrounding the crystalline grains. The term has also been used as a synonym for intergranular.



INTERGRANULAR.—(Ger. *Zwischen Körnern liegend oder verlaufend*, Fr. *Intercellulaire*.)

Between the grains.

INTERPENETRATION.—(Ger. *Durchdringung Interp.*, Fr. *Pénétration*.)

The infiltration of more fusible matter between crystalline faces. It is probable that the harmful effects of segregation are due to the natural strength between crystalline faces being destroyed by the interpenetration of weak and brittle substances, such as sulphide and phosphide of iron (Hogg).

ISOMERIC.—(Ger. *Isomer*, Fr. *Isomérique*.)

All bodies of identical molecular weight were originally termed isomeric; but it is now found convenient (Thorpe) to restrict this term to bodies which not only possess identical molecular weights, but are also of similar chemical type, and possess different physical and chemical properties.

ISOMORPHOUS.—(Ger. *Isomorph*, Fr. *Isomorphe*.)

A term applied to crystals exhibiting similarity in form.

According to Bauerman, it is, in the strict literal sense, only applicable to such substances as are cubical in crystallization. It is, however, largely used in describing substances which crystallize together to form a homogeneous whole, and even although the two or more constituents do not crystallize in the same form. Charpy has described as isomorphous certain crystalline forms which gradually change in composition from the inside outwards. (See "Mixed Crystals.") The term isomorphic mixture is used for such bodies as crystallize together to a homogenous whole.

JOG.

A term used by Professor Howe to express the sudden but limited extension which certain metals undergo at different tenacities. The term was derived from the peculiar character of the curve obtained when the metal is subjected to tension.

Under appropriate conditions, iron and steel of certain classes, if they have previously been neither worked cold nor hardened by quenching, give string diagrams with a well-marked bend where serious permanent set first occurs. This bend, of which the shape varies much, is termed by Professor Howe the "jog."

JOGLESS.

A term also used by Howe to describe iron or metals of which the stress strain diagram does not have this particular inflection. "Smooth" might be misunderstood, and is generic where "jogless" is specific.

JOINT.—(Ger. *Fuge*, *Korngrenze*, Fr. *Joint*.)

This has been used to describe the planes of junction of crystalline grains.

LAMELLA.—(Ger. *Plättchen*, *Blättchen*, Fr. *Lamelle*.)

A term used in microscopic petrography for a thin plate. Plates

of graphite in grey pig iron and the plates of cementite in pearlite are lamellæ.

LAMELLAR. — (Ger. *Blättrig*, Fr. *Lamellaire*.)

Divisible into thin plates.

LAMINA. — (Ger. *Blättchen*, Fr. *Lame*.)

A thin leaf-like plate.

LANCEOLATE. — (Ger. *Lanzettlich*, Fr. *Lancéolé*.)

Lance-shaped; tapering gradually. This term might be discarded.

LAP. — (Ger. *Falz*, Fr. *Crique*.)

A lap is caused by careless hammering, or by badly proportioned grooves in rolls, or by careless rolling, or projections on the ingot. A portion of the iron or steel is folded over on itself, the walls are oxidized, and cannot unite.

LATTICE STRUCTURE. — (Ger. *Gitterförmig Struktur*, *Netzwerk*, Fr. *Treillis*.)

A structure developed on etching Hadfield's manganese steel and some other metals and alloys.

LENTICULAR. — (Ger. *Linsenförmig*, Fr. *Lenticulaire*.)

Shaped like a lens.

LIQUATION. — (Ger. *Saigerung*, Fr. *Liquation*.)

The flowing out from partially solid metals or alloys of a portion of the mass still fluid.

LUSTRE. — (Ger. *Glanz*, Fr. *Éclat*.)

A term used in describing the character of the reflections obtained from the fractured surfaces of minerals and rocks. There are several kinds of lustre — metallic, adamantine, greasy, vitreous, resinous, pearly, silky, etc.

ADAMANTINE. — (Ger. *Diamant*-, Fr. *Adamantin*.)

GREASY. — (Ger. *Fett*-, Fr. *Gras*.)

METALLIC. — (Ger. *Metall*-, Fr. *Métallique*.)

PEARLY. — (Ger. *Perlmutter*-, Fr. *Nacré*.)

RESINOUS. — (Ger. *Harz*-, Fr. *Résineux*.)

SILKY. — (Ger. *Seiden*-, Fr. *Soyeux*.)

VITREOUS. — (Ger. *Glas*-, Fr. *Vitreux*.)

WAXY. — (Ger. *Wachs*-, Fr. *Cireux*.)

MACLES. — (Ger. *Zwillinge*, Fr. *Macles*.)

A synonym of twin crystals.

MACROSCOPIC. — (Ger. *Makroskopisch*, Fr. *Macroscopique*.)

A term used in contradistinction to microscopic, to imply that the character in question is visible to the naked eye. Syn. Megascopic.

MARGARITE. — (Ger. *Margarit*, Fr. *Margarite*.)

The name given by Vogelsang (*Die Krystalliten*, page 19) to the linear arrangement, like strings of beads, assumed by crystallites. Osmond and others have used the term to describe the same arrangement of crystallites in metals and alloys.

MARTENSITE. — (Ger. *Martensit*, Fr. *Martensite* — Osmond.)

This constituent imparts the characteristic structure to polished and etched hardened steels. It has the appearance of a system



of interlacing rectilinear fibres, frequently arranged so as to form triangles. The photograph by Heyn of Charlottenburg of 1 per cent. carbon steel clearly shows this structure, as also do photographs by Osmond of steel containing 0.45 per cent. carbon. It is very fine in 1 per cent. carbon steel and coarse in steel with 0.45 per cent. carbon. According to Osmond, martensite corresponds (1) from a physico-chemical point of view, to the maximum amount of allotropic iron and of hardening carbon that may be retained by very sudden cooling in carburized irons in which the carbon content of the mixed crystals does not exceed about 1.10 per cent. before quenching; (2) from a mechanical point of view, to the maximum of hardness corresponding to such carbon content. In practice, martensite is produced by quenching small samples in cold water.

Arnold suggests that the term should be used for hardened steel containing the same amount of carbon as pearlite.

The composition of martensite is very indefinite. Baron Jüptner imagines it to be a solution of carbide of iron in iron, while Campbell gives its composition as  $C_2Fe_3$ .

**MASSIVE.**—(Ger. *Massiv*, Fr. *Massif*.)

A term used in contradistinction to "stratified." Metallographers have used the term for free cementite as distinguished from that in pearlite. The term does not, however, imply homogeneity.

**MATRIX.**—(Ger. *Grundmasse*, Fr. *Magma*.)

This term appears to embrace the terms mother-substance and eutectic.

**MEGASCOPIC.**—(See "Macroscopic.")

**METALLOGRAPHY.**—(Ger. *Metallographie*, Fr. *Métallographie*—Osmond.)

In a most general sense is a description of the structure of metals and their alloys, but may also be described as the science that deals with the composition, constitution, structure, and physical properties of metals and alloys, but does not include the art of metallurgy. To that branch of the science dealing with iron and its alloys Baron Jüptner has applied the name of *Siderologie*.

**METALLURGY.**—(Ger. *Hüttenkunde*, Fr. *Métallurgie*.)

The art of working in metals.

**MELTING-POINT CURVE.**—(Ger. *Schmelzpunktskurve*, Fr. *Courbe des points de fusion*—Jüptner.)

This is identical with "freezing-point curve."

**MIXED CRYSTALS.**—(Ger. *Mischkrystalle*, Fr. *Cristaux mêlés*.)

A term used for two or more substances which crystallize together into a homogeneous whole. The homogeneous solution of two or more crystallized bodies while in a solid state. From the point of view of the "phase doctrine" these form a single phase.

**MOTHER-LIQUOR.**—(Ger. *Mutterlauge*, Fr. *Liqueur-mère*.)

During the process of congelation of any system consisting of several substances in a state of solution, a portion of the solution remains in a fluid state after the separating out of solid crystals;

this liquor is termed "mother-liquor." In systems consisting of two constituents with a eutectic point, the composition of this liquor approximates as congelation proceeds to that of the eutectic mixture, and quite attains to this composition before the whole mass passes into the solid state.

NATURAL HARDNESS. — (Ger. *Naturhärte*, Fr. *Dureté naturelle*.)

The original degree of hardness possessed by steel before quenching above  $A_{r1}$ .

NEEDLE. — (Ger. *Nadel*, Fr. *Aiguille*.)

(See "Acicular.")

NON-EUTECTIC CEMENTITE.

That part of the cementite in iron and steel which is external to the pearlite in high carbon.

ORIENTATION. — (Ger. *Orientierung*, Fr. *Orientation*.)

The relative direction of the axes of crystals, or of the axes of elasticity in two or more crystals, or the relative position of these axes with regard to a certain surface or line; for instance, the polished surface or a cleavage plane, etc.

OSMOTIC PRESSURE. — (Ger. *Osmotischer Druck*, Fr. *Pression osmotique*.)

When two liquids are separated by the intervention of a porous diaphragm, a flow of liquid takes place from one side to the other, or sometimes an unequal flow of the two liquids in opposite directions. This phenomenon is termed "osmose," and the pressure exerted by a dissolved substance in its solution is termed its osmotic pressure.

OVERHEATED. — (Ger. *Ueberhitzt*, Fr. *Surchauffé*.)

Applied to steel that has been heated too far and not quite burned.

PEARLITE. — (Ger. *Perlit*, Fr. *Perlite*, U. S. *pearlyte*.)

A term first applied by Professor Howe as a synonym for the pearly constituent discovered by Dr. Sorby of Sheffield. It consists of alternating plates of cementite and ferrite, or sorbite, or grains of cementite embedded in ferrite or sorbite.

Baron Jüptner considers that the cementite of the pearlite consists chiefly of the carbide  $C_4Fe_{12}$ , but that the sorbite perhaps contains  $C_3Fe_9$ . This corresponds to E. D. Campbell's data.

PEARLY CONSTITUENT. — (Sorby.)

Synonym pearlite.

PHASE. — (Ger. *Phase*, Fr. *Phase*.)

In the sense adopted by J. W. Gibbs in his "Physical Chemistry." It is excellently rendered and described with apt explanations by Le Chatelier in his memoir "La Loi des Phases," *Revue Générale des Sciences*, 10 Année, No. 20, 1899, ii., 1st paragraph. A phase is defined as a mass chemically or physically homogeneous, or as a mass of uniform concentration. The state of a phase is completely determined if the pressure and temperature, together with the chemical potential of its components, be known.



PHASE DOCTRINE. — (Ger. *Phasenlehre*, Fr. *La doctrine des phases*.)

The doctrine or teaching relating to the phase rule. An expression used by Professor Roozeboom.

PHASE RULE. — (Ger. *Phasenregel*, Fr. *Loi des phases*.)

A rule for finding the number of phases that can exist in a system containing a certain number of components, and having a certain number of degrees of freedom. It connects together the number of components, degrees of freedom, and possible phases in equilibrium (Gibbs).

The phase rule states that in a system of  $n$  components there can exist in equilibrium  $n + 2$  phases if the system be nonvariant,  $n + 1$  if it be monovariant,  $n$  if it be divariant, and so on (Stansfield).

In physical chemistry the term "phase" is understood to mean homogeneous particles which occur within a body, and are separated by dividing surfaces from the other parts of that body. For example, ice, water, and steam. Examples of phases occurring in steel are ferrite, martensite, and cementite, while pearlite contains two or three phases—ferrite, cementite, and sorbite (Jüptner).

PIPE. — (Ger. *Lunker*, *Schwindungshohlraum*, Fr. *Retassure*.)

A pipe is the cavity formed in the central upper part of the ingot.

POLISH ATTACK. — (Ger. *Reliefpolieren*, Fr. *Polissage en bas-relief* — Osmond.)

When alloys contain two or more constituents of different degrees of hardness, by long continued friction on a soft polishing surface they are worn away in different degrees, the hardest constituent being least acted upon, the softest the most. The surfaces have all the appearance of having been etched.

POLISH ETCHING. — (Ger. *Actzpolieren*, Fr. *Polissage attaque*.)

The process of slightly acting upon polished surfaces of metals by friction on parchment moistened with a solution of nitrate of ammonia, so as to reveal the structure. The sorbite, troostite, and martensite are colored, but ferrite and cementite remain white.

POLISHING. — (Ger. *Polieren*, Fr. *Polissage*.)

The process of preparing perfectly bright and smooth surfaces free from even microscopic scratches.

POLYMORPHISM. — (Ger. *Polymorphismus*, Fr. *Polymorphisme*.)

The property possessed by some substances of assuming two or more forms of molecular structure.

PRISMATIC. — (Ger. *Prismatisch*, *Säulenförmig*, Fr. *Prismatique*.)

As applied to alloys, a term indicating that the crystals present occur in columnar forms giving lath-shaped sections.

PSEUDOMORPHOUS. — (Ger. *Pseudomorph*, Fr. *Pseudomorphe*.)

A false form. Applied to minerals possessing a crystalline form other than their own, which they have obtained by the displacement or alteration of the original mineral. Osmond has recently used the term for the crystalline grains in iron where their forms are determined by mutual interference.

RECALESCENCE. — (Ger. *Recalescenz*, Fr. *Recalescence*. — Barrett.)

A term used to express the phenomenon of the evolution of internal heat which occurs when iron and steel cool through the critical zones.

RETICULATED. — (Ger. *Netzförmig*, *Netzartig*, Fr. *Réticulé*.)

Having the appearance of network; traversed by two sets of parallel lines.

ROTATION EFFECT.

An expression used to describe lighting up and darkening of the crystalline grains in the etched surfaces of polished metal when rotated before oblique rays of light (Heycock and Neville). Professor Ewing and Mr. Rosenhain describe the same as "selective effect of oblique light."

SCLEROMETER. — (Ger. *Sklerometer*, Fr. *Scléromètre*.)

An instrument designed by Mr. T. Turner to determine the relative hardness of metals.

SCORIACEOUS. — (Ger. *Schlackenartig*, *Schlackig*, Fr. *Scoriacé*.)

Having the cindery appearance of scoriæ. A suitable term to use when the exact nature of the substance is not known when detected as inclusions in metals and alloys.

SCORIÆ. — (Ger. *Schlackenartiger Körper*, Fr. *Scories*.)

Cinder or slags from metallurgical processes.

SEAM. — (Ger. *Naht*, Fr. *Couture*.)

A seam is a longer or shorter effect, caused by a blowhole which working has brought out to the surface and not eliminated. It usually, or always, runs in the direction of working. Seams are distinguished from laps by not being continuous; they are usually only an inch or two in length (Metcalf). (Syn. Roke.)

SEGREGATION. — (Ger. *Ausscheidung*, *Saigerung*, Fr. *Ségrégation*.)

A term applied to the more fusible parts of metals and alloys which are last to freeze, and are driven into certain local centres by the metal which first crystallizes. Segregates are generally found near the centre of the castings, and are most pronounced in very large masses.

SHORT. COLD, RED.

COLD-SHORT. — (Ger. *Kaltbrüchig*, Fr. *Cassant à froid*.)

Cold-short steel is weak and brittle when cold.

RED-SHORT. — (Ger. *Rothbrüchig*, Fr. *Cassant à chaud*.)

Steel is red-short when it is brittle on working at a red heat.

SKELETON CRYSTALS. — (Ger. *Krystallskelette*, *Gestrickte Formen*, Fr. *Réseau du cristal*.)

The incipient forms of crystallization in which some metals or metallic substances appear in cast metals and alloys.

SLAG INCLUSIONS. — (Ger. *Schlackeneinschlüsse*, Fr. *Scories enfermées*.)

Particles of slag enclosed in a metal.

SLIP-BANDS. — (Ewing and Rosenhain.)

A microscopic appearance in smooth surfaces of metals after straining. Caused by slips along the cleavage or gliding planes of the crystalline grains.



SOLID SOLUTIONS. — (Ger. *Feste Lösungen*, Fr. *Solutions solides* — Van 't Hoff.)

A solid solution is a homogeneous mixture of two or more substances in the solid state. In metals no one has yet worked at non-crystalline mixtures, and solid solutions of metals when crystalline are solid "isomorphous mixtures" or "mixed crystals" (Sir W. Roberts-Austen).

SOLIDIFIED SOLUTION. — (Ger. *Erstarrte Lösung*, Fr. *Solution congelée*.) (See "Congealed Solution.")

SOLUTION PLANE. — (Ger. *Lösungsfläche*, Fr. *Direction de solution*.)

The name proposed by Von Ebner in 1814 for a plane in a crystal along which it is most easily attacked when submitted to chemical action. Such solution planes are commonly observable on acid etched surfaces of iron.

SORBITE. — (Ger. *Sorbit*, Fr. *Sorbite* — Osmond.)

Sorbite is one of the constituents of steel which is a transition between the troostite and pearlite state.

Jüptner believes that in all probability this is a solution of  $\text{Fe}_3\text{C}_2$  in Fe, and perhaps may be identical with troostite.

It might almost be called "un-segregated pearlite." Between it and troostite or sorbite there exists no sharp line of demarcation. Sorbite is obtained irregularly by a cooling sufficiently slow to allow the transformation to proceed far and sufficiently rapid to produce an imperfect separation of ferrite and cementite. In practice these conditions are more or less fulfilled in the cooling in air of small samples, in quenching in cold water towards the end of the recalescence, in quenching in molten lead, in certain "double trempes," or in reheating (tempering) martensite to a blue color.

On etching the polished steels by the polish attack, or by iodine and dilute acids, sorbite is colored brown.

SPHERULITIC STRUCTURE. — (Ger. *Sphärolithische Struktur*, Fr. *Structure globulaire*.)

Peculiar to vitreous rocks, and in eutectics of alloys rapidly cooled, and so called because of the small spherular bodies (spherulites) they contain. The latter, viewed under a microscope, are seen to possess a divergent fibrous structure. This structure is common in the rapidly cooled eutectics of lead — tin, antimony — lead, and gold — lead.

STELLATE. — (Ger. *Sternförmig*, Fr. *En étoile*.)

Radiating from a centre as in some of the copper-tin alloys rich in tin, in which the definite compound  $\text{CuSn}$  crystallizes in stellate or star-like forms. This term might be discarded.

SURFACE TENSION. — (Ger. *Oberflächenspannung*, Fr. *Tension superficielle*.)

The surface of a liquid has greater cohesion than any layer of the liquid in the interior. The surface is, as it were, stretched by an elastic skin. This surface tension is greater, the greater the cohesion of the liquid.

SURFUSION. — (Ger. *Ueberschmelzung*, Fr. *Surfusion*.)

A transitory liquid state at temperatures below the 'normal freezing-point.

TEMPER. — (Ger. *Kohlungsgrad*, Fr. *Degré de recuit*.)

Used by the steelmaker, it means the quantity of carbon present (Metcalf). It is low temper, medium or high, or number so-and-so by his shop-numbers. Used by the steel-user or by the temperer, it means the color to which hardened steel is drawn—straw, brown, pigeon-wing, blue, etc., etc. Or it is the steelmaker's measure of initial hardness, and it is the steel-user's measure of final hardness.

LOW TEMPER. — (Ger. *Weich*.)

MEDIUM OR HIGH TEMPER. — (Ger. *Mittelhart*, Fr. *Hart*.)

STRAW YELLOW. — (Ger. *Strohgelb*, Fr. *Jaune paille*.)

BROWN. — (Ger. *Braun*, Fr. *Brun*.)

VIOLET. — (Ger. *Violett*, Fr. *Violet*.)

BLUE. — (Ger. *Blau*, Fr. *Bleu*.)

LOW TEMPER STEEL. — (Ger. *Weiches Flusseisen*.)

MEDIUM TEMPER STEEL. — (Ger. *Mittelhartes Flusseisen*.)

HIGH TEMPER STEEL. — (Ger. *Harter Stahl Flusseisen*.)

TENACITY. — (Ger. *Zugfestigkeit*, Fr. *Ténacité*.)

The resistance to rupture presented by a substance whose elastic limit has been exceeded.

TEMPERING COLORS. — (Ger. *Anlauffarben*, *Anlassfarben*, Fr. *Couleurs de recuit*.)

These are the colors which appear on iron and steel surfaces when slightly heated in air.

TROOSTITE. — (Ger. *Troostit*, Fr. *Troostite*.)

This constituent, according to Osmond, is contained in steels of various carbon contents on quenching at certain temperatures. It is revealed by polishing the steel on parchment, moistened with a solution of liquorice root or nitrate of ammonia. It readily assumes a brown color by such treatment. It is relatively softer and more rapidly acted upon by acids than martensite. Its composition and molecular construction has not been determined.

This is easily recognizable on etching with hydrochloric acid (1 cubic centimetre) in alcohol (100 cubic centimetres). It colors dark, while martensite remains entirely unaffected in color. By reason of the troostite being softer than the surrounding martensite, it has not hitherto been obtained in tangible form (Martens and Heyn.)

According to the newest researches on expansive properties by H. Le Chatelier, this is an intermediate formation which occurs on the transformation of martensite into pearlite and *vice versa*. It is very likely a solution of the carbide  $C_3Fe_9$  in iron.

TWINNED. — (Ger. *Verzwillingt*, *Zwillingsbildung*, Fr. *Hémitrope*, *Maclé*.)

A crystal is twinned when two portions of the same individual, or two different individuals, are related to one another according to a definite law.



# THE FREEZING-POINT CURVE OF BINARY ALLOYS OF LIMITED RECIPROCAL SOLUBILITY WHEN MOLTEN\*

By H. M. HOWE

IN the admirable paper of Roberts-Austen and Stansfield, "*La Constitution des Alliages Métalliques*,"† they show a figure which I now reproduce as Fig. 1. We have here an illustration of the danger which Le Chatelier has pointed out, that the very

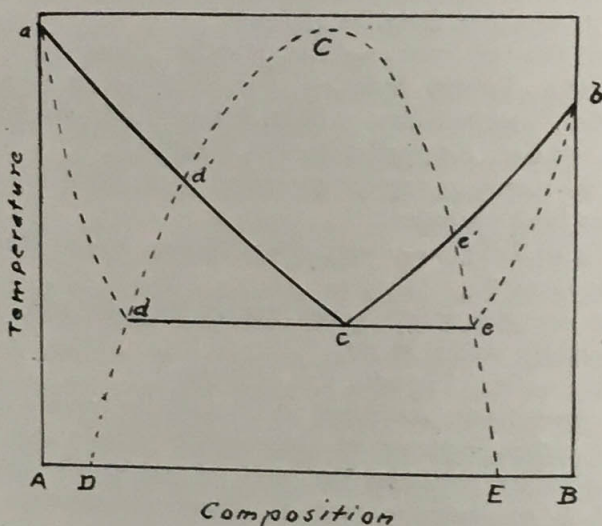


Fig. 1. Roberts-Austen and Stansfield.

convenience of these graphical illustrations may cause them to mislead. This present Fig. 1 might mislead in a way that I will now try to point out:

The fact that the continuation  $d'Ce'$  of the solubility or so-called "critical" curve is here shown as a smooth continuation might lead the reader to suppose that, in case of an alloy of two metals of limited reciprocal solubility when solid, and also limited

\* Received in substantially the present form, excepting the last paragraph, on February 14, 1902.

† Rapports Présentés au Congrès International de Physique Réuni à Paris en 1900, I, 363.

reciprocal solubility when molten, the solubility curve for the molten state would normally and naturally be a smooth continuation of the same curve for the solid state. It is not to be supposed that this was the writers' meaning. The reciprocal solubility for the molten state ought really to be very much greater than for the solid state, so that the curve for the molten state, instead of being a smooth continuation of that for the solid state, should be separated from it by a sharp and long jog like  $f'M$  and  $QJ$  at the upper freezing-point line (Fig. 2).

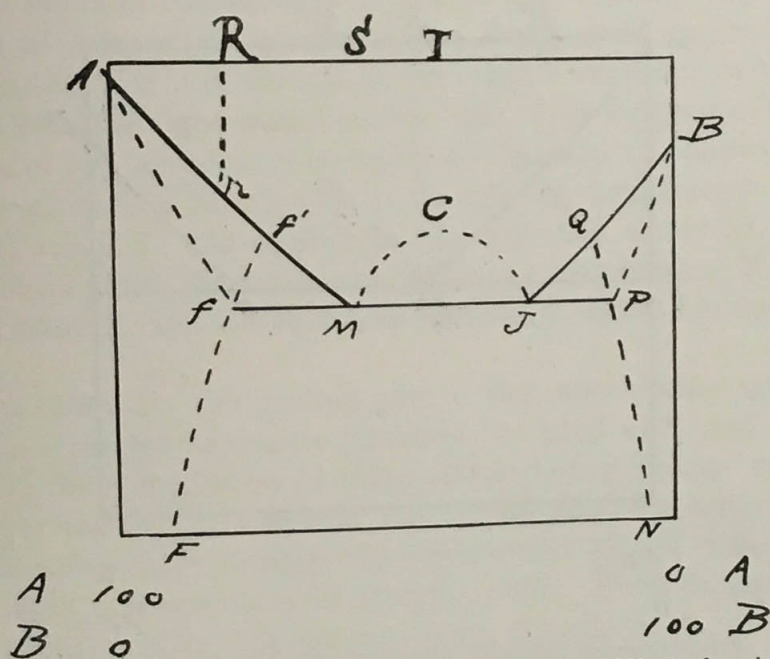


Fig. 2. Freezing-point curve of alloys of two metals of limited reciprocal solubility, both when molten and when solid.

Fig. 1 might also lead the hasty reader to suppose that, in case of limited reciprocal solubility when molten, as shown by  $d'Ce'$ , the freezing-point curve would nevertheless be of the sharp underscored V-type so familiar, or in other words that the lines  $d'C$  and  $e'C$  would actually be present. Now, in point of fact, those lines would be lacking and the shape of the freezing-point curve would be that given in Fig. 2 if, to take the simplest possible case, we assume that the solubility curve for the molten state cuts the two branches  $AM$  and  $BJ$  at the same temperature, as is readily seen on consideration.

For simplicity let us further assume that each of these two



metals, A and B, is absolutely insoluble in the other when solid. In this case the lines Af, Fff', BP and NPQ would disappear, and the curve would be that shown in Fig. 3. Let us test the shape of AMJB by following the freezing of three different alloys, those with R, S and T per cent respectively, of metal B.

*Alloy R.*—This in cooling would begin freezing at *r* with the solidification of metal A and enrichment of the mother metal in metal B, as in the common course of selective freezing; so that the temperature and composition of the mother metal at succes-

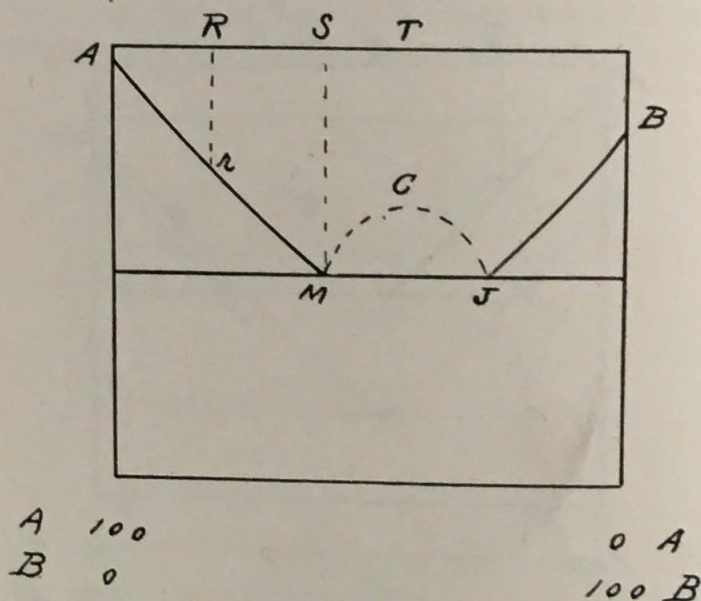


Fig. 3. Freezing-point curve of alloys of two metals of limited reciprocal solubility when molten, but complete reciprocal insolubility when solid.

Case 1: solubility curve cuts both upper branches of the freezing-point curve at the same temperature.

sive instants would be represented by a series of points between *r* and M. In short, the temperature and composition of the mother metal slide from *r* to M. But on reaching M this sliding is arrested, and the mother metal will now freeze without further fall of temperature as the eutectic, breaking up in freezing into separate particles of the two metals A and B. The reason why selective freezing should cease on reaching M, and that the mother metal should there freeze without further selection, i.e., as a whole, is of course the same which leads to the freezing of the eutectic as a whole in any ordinary alloy as soon as the eutectic composition

and freezing point are reached. At each instant during the selective freezing from  $r$  to  $M$  the mother metal has been at its then freezing point, and it has avoided freezing as a whole by ejecting from itself part of the excess or solvent metal  $A$ , and so becoming enriched in the dissolved metal  $B$ , and thereby acquiring a lower freezing point. This can go on only so long as selective freezing is capable of leading to an alloy of a lower freezing point. Now this possibility ceases when the temperature and composition of the mother metal reach  $M$ , for that is by assumption the freezing point of the most fusible alloy of the series. Indeed, no possible shifting of composition of the mother metal could further defer freezing. Moving it to the left would make it still more infusible; moving it to the right would simply split it up into a mechanical mixture of (1) an alloy of composition  $M$  and (2) another alloy of composition  $J$ , both infusible at the existing temperature.

(2) *Alloy S*. On cooling to  $M$ , this alloy would be in the condition in which the mother metal was at temperature  $M$  in the case of alloy  $R$ , and would freeze there as a whole for the same reason.

(3) *Alloy T*. On cooling past  $C$  the molten alloy splits up into two saturated conjugate solutions (1) of  $B$  in  $A$ , and (2) of  $A$  in  $B$ ; the composition of these shifts during further cooling, following the curves  $CM$  and  $CJ$ , till on reaching temperature  $M$  these alloys have respectively composition  $M$  and composition  $J$ , and each is therefore at its freezing point. Each therefore begins freezing,  $M$  by the deposition of crystals of  $A$ , and  $J$  by deposition of crystals of  $B$ . The temperature cannot sink below  $M$  until the whole has frozen, because there is no single alloy of these two metals possible at this or any lower temperature with composition intermediate between  $M$  and  $J$ , and because the existing temperature is at or below the freezing point of  $M$  and every alloy at its left, and that of  $J$  and every alloy at its right. This completion of the freezing at  $M$  follows also from the phase rule, for once the freezing of either  $M$  or  $J$  with separation of crystals of  $A$  or  $B$  begins, the system is invariant, there being thus three phases (alloys  $M$  and  $J$  molten, and metal  $A$  or  $B$  solid), with only two constituents  $A$  and  $B$ . Because of this invariance, the temperature cannot (in equilibrium) sink, until the number of phases has fallen to two, metals  $A$  and  $B$ , by the elimination of  $M$  and  $J$ . Only thus will the system become monovariant:



Degree of liberty = No. of constituents (2) + 1 — No. of phases (2) = 1.

CASE 2. If, as in the diagram of Roberts-Austen and Stansfield, the solubility curve cuts the two branches of the V or upper-freezing-point curve at different temperatures, then the freezing-point curve assumes the peculiar shape shown in Fig. 4, assuming as before for simplicity that when solid each metal is absolutely insoluble in the other. Here the upper freezing points for alloys containing between 70 and 100 per cent of A are rep-

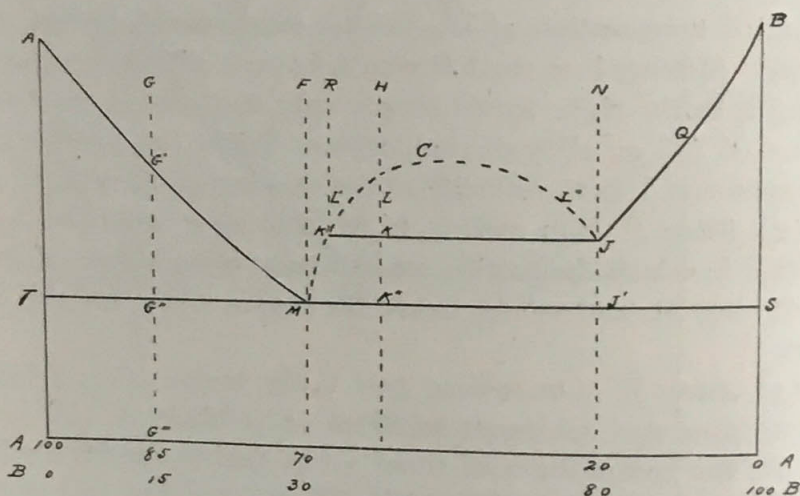


Fig. 4. Freezing-point curve of alloys of two metals of limited reciprocal solubility when molten, but complete reciprocal insolubility when solid.

Case 2: solubility curve cuts the upper branches of the freezing-point curve at different temperatures.

resented by AM, and those for alloys between 0 and 20 per cent of A by BJ, and the lower freezing points for these two regions will be on the lines TM and J'S. For alloys of intermediate composition, containing between 20 and 70 per cent of A, the upper freezing point is JK'M, and the lower freezing point is J'M.

To test this let us follow the cooling of a few different alloys containing respectively G, F, H, and Q per cent of metal B.

*Alloys G and F.* The shape of the freezing-point curve at M and at the left of M is shown by the same reasoning that established the corresponding part of Fig. 3. (See alloys R and S of Fig. 3.)

*Alloy H* is a molten solution of B in A, which on cooling

to the critical curve becomes saturated, and on passing below the critical curve splits up into two conjugate saturated molten solutions (1) a large quantity of a solution of B in A, and (2) a small quantity of the solution of A in B. When the temperature has fallen to L, alloy 1 will contain  $L'$  per cent of B dissolved in  $100-L'$  per cent of A, and alloy 2 will contain  $100-L''$  per cent of A dissolved in  $L''$  per cent of B. When the temperature falls to K, alloy 1 will contain  $K'$  per cent of B dissolved in  $100-K'$  per cent of A, and alloy 2 will contain  $100-J$  per cent of A dissolved in  $J$  per cent of B. In short, the composition of these two alloys will be  $K'$  and  $J$  respectively. Alloy J, however, will now be at its freezing point, and without further fall of temperature it will begin freezing.

In the freezing which now takes place alloy J should in effect deposit only metal B, as we see on trying to imagine the contrary. Suppose that the first flake of alloy J in freezing at J does so without selection, *i. e.*, that it deposits a flake of composition J. As metals A and B by assumption are insoluble in each other, this first flake must (if equilibrium be reached), break up into particles of pure B and separate particles of pure A. But these pure particles of A, exposed as they are in the very act of freezing to alloy  $K'$ , which is a saturated solution of B in A, should immediately remelt and reënter alloy  $K'$ , since the alloy which their re-melting would create is fusible at the existing temperature because it has more than  $K'$  per cent of A, *i. e.*, because its composition lies to the left of  $K'$ . This re-melting then would give us two molten alloys, alloy J, and an alloy with composition to the left of  $K'$ . But as the mean composition of the mother metal would still be between J and  $K'$  it would immediately rearrange itself into a mechanical mixture of two alloys of composition J and  $K'$  respectively. The effect of the freezing and re-melting which have taken place at temperature J thus would be to eliminate from the molten mass a small quantity of metal B, and so to shift the composition of the mother metal from K toward  $K'$ . But for any composition at temperature J and any point between  $K'$  and J, the mother metal will be a mixture of alloy  $K'$  and J, so that as regards further freezing of alloy J we have exactly the same condition that we had before the first flake began to freeze. Following this, flake by flake, we see that what really happens is that metal B begins freezing



out at constant temperature, and that the composition of the mother metal progressively travels from K to K', which means that the quantity of alloy J is constantly diminishing and that of alloy K' is constantly increasing until, when the composition reaches K', the whole of alloy J has been removed by the freezing out of the metal B, and the mother metal now consists exclusively of alloy K'. The temperature will thus remain constant during this freezing out of alloy J.

Indeed, this constancy of temperature during the freezing out of alloy J follows from the phase rule. For, when the first flake of metal B has frozen, the system has become invariant, having three phases, alloy J and alloy K' molten, and solid metal B; the temperature therefore cannot further descend until one of these phases has been eliminated. That to be eliminated is clearly alloy J, through the progressive freezing out of metal B, until the residual molten mother metal has been reduced to a single member, alloy K'. The system then becomes monovariant, since it is di-phase, molten alloy K' and solid metal B, and contains two constituents, metals A and B.

Now that the mother metal consists solely of an alloy of composition K' the temperature will again begin to fall. As it falls, the mother metal becomes super-saturated with metal B, and consequently the excess of metal B keeps freezing out, and this continues until the temperature falls to M, so that during this cooling the temperature and composition of the mother metal, alloy I, simultaneously slide along the solubility curve from K' to M. Then the mother metal will freeze unselectively,\* like any

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\* That the freezing at M will be unselective is seen readily on trying to suppose the opposite, i.e., that it is selective. Selective freezing can occur only through a selection which gives to the mother metal a freezing point lower than the existing temperature. But such selection cannot occur through the freezing out of part of metal B, because that would move the composition to the left and would make the mother metal more infusible. The freezing on the other hand cannot be made selective in the sense that metal A freezes out, because the mother metal is already a saturated solution of 30 per cent of B in 70 per cent of A. Reducing the quantity of A would give a solution supersaturated with B, which, assuming for the instant that it was momentarily molten, would split up into a mixture of solutions containing respectively M and J (70 and 20) per cent of A. But each of these would forthwith freeze, M because the temperature is at its freezing point, J because the temperature is below its freezing point.

eutectic, simply because no selective form of freezing can postpone complete freezing. In freezing the eutectic will, like any other, split up into its components A and B, assumed to be insoluble in each other when solid.

Thus for composition H we have two freezing points: K and K''. The same would be true of any alloy of composition between R and N. For alloys between R and F, the upper freezing point will be on the line K'M and the lower freezing point will be on the line MJ'.

This then gives us the freezing-point curves between F and N, viz., for the upper freezing point MK'J and for the lower freezing point MJ'.

*Alloy Q.*—The series of alloys between N and B will have for the upper branch of the freezing-point curve BJK'M, and for the lower branch J'S. Taking for instance alloy Q, it will begin freezing selectively by the solidification of the excess or solvent metal B; and the composition and temperature of the mother metal will, as in all like cases, slide from Q to J. But as the temperature starts to descend below J, the composition of the mother metal crosses the solubility curve, and the mother metal therefore splits up into a mechanical mixture of two alloys of compositions K' and J respectively. Of these J is already at its freezing point, and therefore freezes. But, exactly as in all cases of the freezing of binary alloys of complete reciprocal insolubility when solid, and as explicitly set forth in the second paragraph of our consideration of alloy H, it is only metal B which will freeze out, so that the composition of the mother metal begins to slide to the left along JK'. But we now have the same condition of affairs as in the freezing of alloy H, viz.: an invariant system consisting of (1) alloy J, and (2) alloy K', both molten, and (3) metal B solid; and, for the same reason as in case of alloy H, the temperature of the mother metal remains constant until its composition reaches K', when, by the complete elimination of alloy J, it has become monovariant. Then the temperature and composition as before slide from K' to M, where the eutectic freezes as a whole, i.e., without further selection.

The branch TMJ'S will extend clear to the right-hand side of the diagram, because, thanks to the assumed complete insolubility of A in B when solid, only pure metal B freezes out as the temperature and composition of the mother metal slide along the

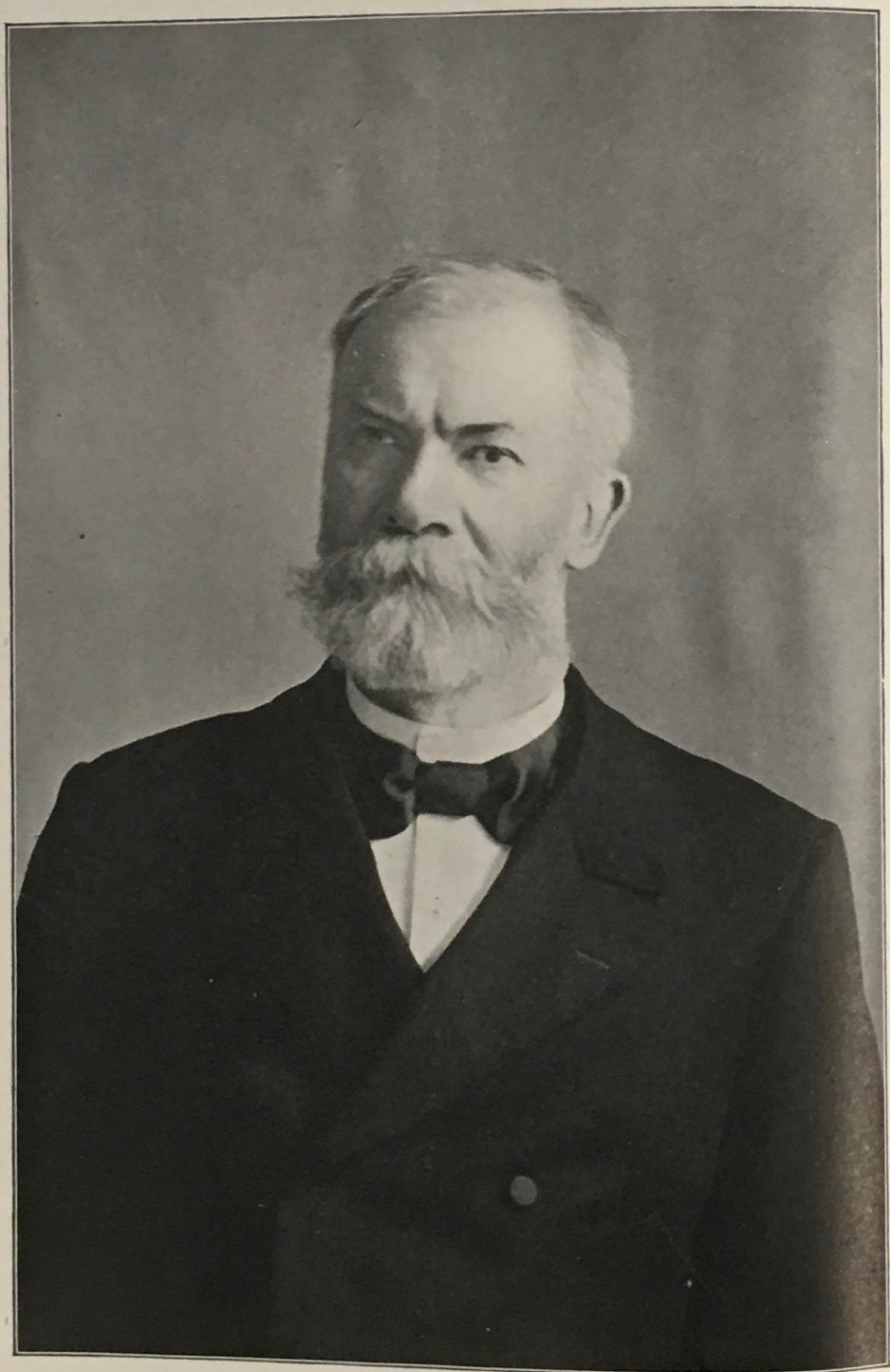


lines BJ, JK', and K'M. This freezing out of B and enrichment of the mother metal in A, having nothing to arrest them, will continue until the mother metal reaches composition M with its 70 per cent of A; so that a finite quantity of the eutectic M will form no matter how small the initial quantity of A, and this eutectic, freezing when temperature and composition M are simultaneously reached, will give rise to the lower freezing point on the line TS.

It is assumed throughout this note that the conditions are those of complete equilibrium, and the influence of lag and indeed of every disturbance of equilibrium is neglected.







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## DIMITRY TSCHERNOFF

THE younger generation of steel metallurgists rightly look upon Professor Tschernoff as the originator of the scientific investigation of the properties and structure of steel. His memoirs describing the results of his admirable work, published as early as 1868, will long remain classical and will long serve as models of clearness and logic to students in metallurgy.

Dimitry Tschernoff was born in St. Petersburg October 8, 1839. After graduating from the Institute of Technology of that city in 1858, he studied three years in the department of mathematics of the University of St. Petersburg. He was then appointed tutor at the University, and taught algebra and geometry for a few years. In 1863 he became assistant librarian at the Institute of Technology.

In 1866 Professor Tschernoff left the field of pure science to accept an offer of Colonel Oboukhoff to take charge of the department of heavy forgings at the Oboukhoff Steel Works, near St. Petersburg. It is during his connection with these works that he conducted his famous researches dealing with the influence of thermal treatment upon the structure of steel and with the relation between the structure and the physical properties. His new but sound theory was applied to the manufacture of steel guns and resulted in immediate and great improvements. He also took an active part in the development of the other depart-



ascertain the definite cause of the weakness, whether chemical or physical.

Up to the present time about 200 defective rails have been examined. In some cases complete chemical analyses have been made; in others the loose, coarse-grained fracture or other physical character — such as piping — showed the cause at a glance, and in still others a rather elaborate investigation was necessary to prove the matter to a certainty.

It is evident, of course, that condition and arrangement of track has an important bearing upon durability of rails, and this matter, as we all know, has been worked out exhaustively by Dr. P. H. Dudley.\* Our experience, however, indicates that in the large majority of cases with normal track conditions, short life of rails is attributable to the character of the steel, and is seldom found where toughness, strength and solidity exist.

In a general way, the results of analysis in this investigation have merely confirmed the previous opinion, and have proved beyond question that specifying chemical composition alone ensures neither a durable rail nor often a bad rail. We found, however, from the start a marked difference in the structures, and note the following general characteristics in the rails which gave defective service:

(a) Coarse regular granular structure, Figs. 1 and 8 (50 diameters).

(b) Excess of foreign matter, such as oxides, slag and enclosed gas, Fig. 2 (reduced 50 per cent).

Either characteristic resulted in relatively poor service.

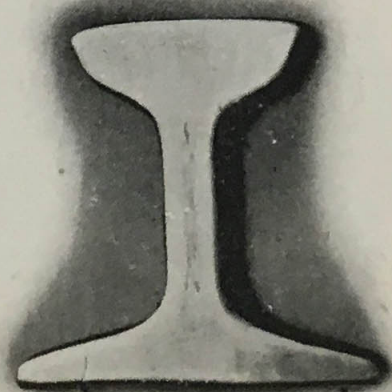
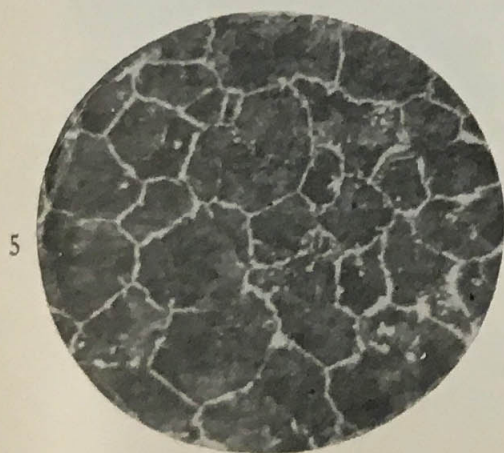
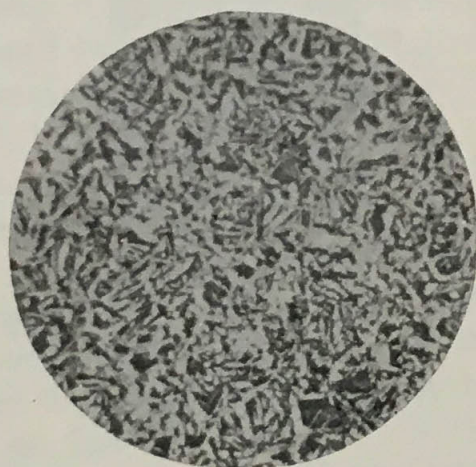
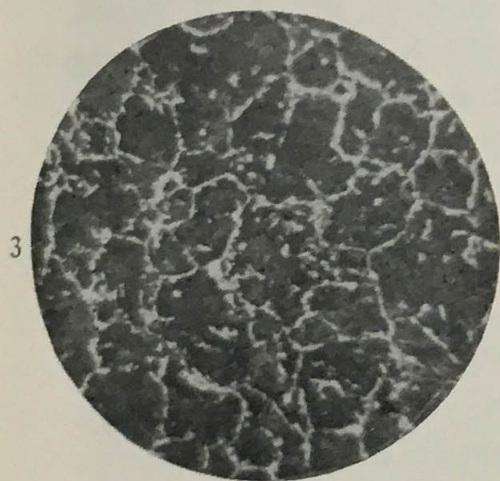
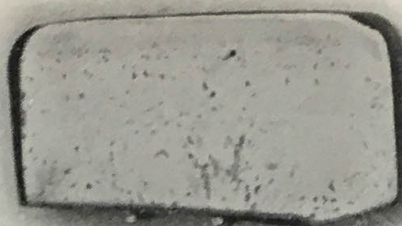
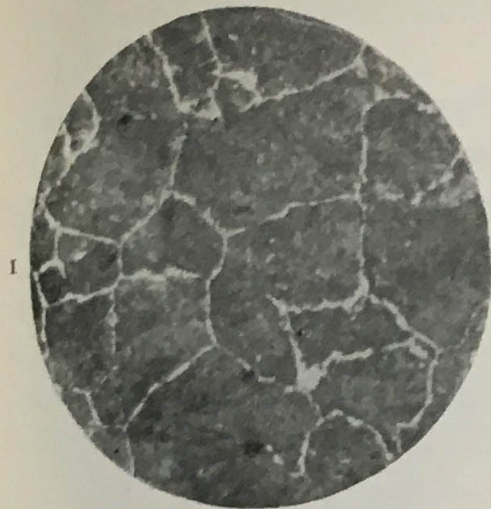
On the other hand, in rails of the same general composition giving satisfactory service, we have found (c) a generally fine, interlocking, broken up, granular form, with

(d) Relative freedom from foreign matter. Fig. 3, which will be referred to again, represents a coarse example of this finer structure, and Fig. 4 shows a characteristic structure of the head, reduced 54 per cent, showing freedom from foreign matter.

Upon comparing the above results with those obtained in our mill inspections we found complete accordance, for the rails of the character *a - b* proved exceedingly fragile under the drop-test of 2,000 pounds falling 20 feet. A section of rail, Fig. 2, from center of head, gave an elongation of only 2 per cent in a 2-inch

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\* *Bulletin International Railway Congress*, XIV, No. 6, June, 1900.





section, with tensile strength of 118,000 pounds per square inch. The analysis of these rails varied from:

Carbon, .55 to .63 per cent; phosphorus, .075 to .104 per cent; manganese, .75 to 1.20 per cent, and sulphur, .034 to .07 per cent.

In contrast to this, rails when of the structure *c-d*, with the above composition, showed a marked toughness under the drop-test, and rail Fig. 4 stood fourteen blows of the drop without fracture, turning after the first and third blows, and successive odd numbers. The composition was: carbon, .644 per cent; manganese, 1.09 per cent; phosphorus, .081 per cent; sulphur, .076 per cent, and a test piece from a similar rail, center of head, gave an elongation of 13 per cent in 2 inches, with tensile strength of 132,000 pounds per square inch.

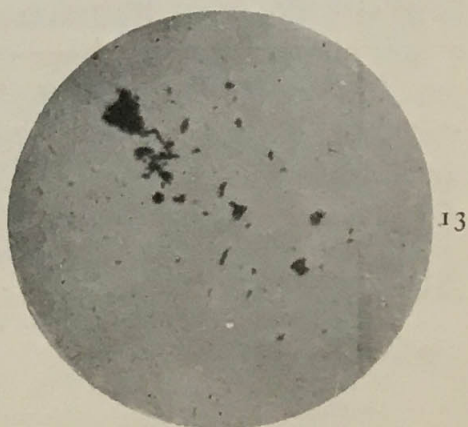
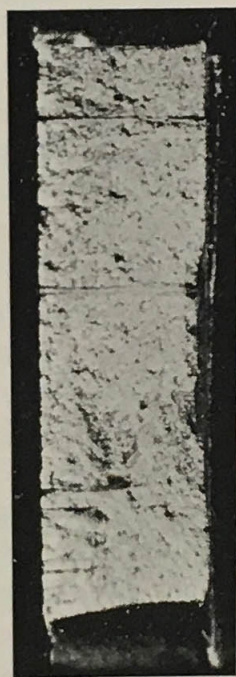
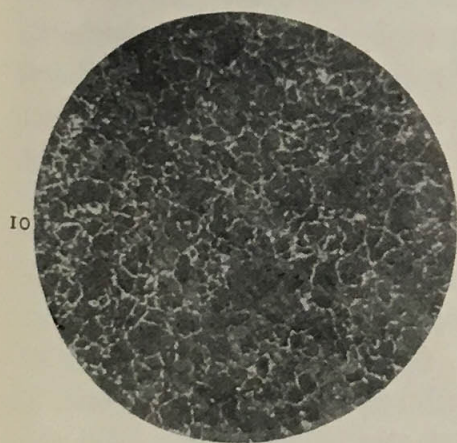
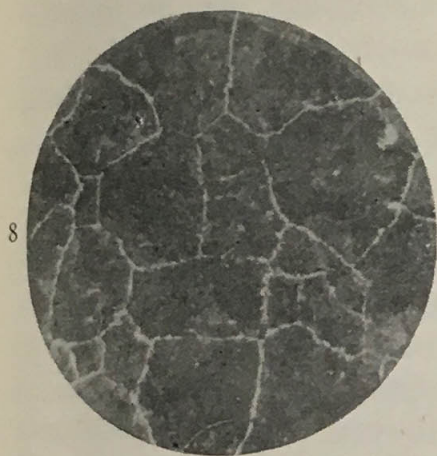
These marked differences were not at all unexpected, for they simply confirm the work of others. Mr. J. E. Stead, for instance, says:\* "It is clear that the junctions (of the grains) are a safeguard, and the more junctions there are, the more reliable will the steel be." And again,† "it would seem to follow, then, that the smaller and finer the grain, the safer the structure. This coincides with the researches of Brinell, Sauveur and others.

Next, in order to find out how these results tallied with those of practical track service, we made a comparison upon a lot of about fifty rails which fractured in track, each having had a life of less than five years. They came from different points upon our lines, and had approximately the composition stated above. A number of different mills were represented, but all of the rails could be grouped under the head of "coarse-grained," or of "finer grain," and upon figuring the fractures in each class to a basis of equal tonnage during the five years, we found an average of fifteen fractures of the coarse-grained material to one fracture of the relatively finer-grained, and in each case of fracture of the latter, we found that the weakness was due to pipes in the steel—defective cropping. This comparison, of course, applies strictly only to actual fracture, but it is well known that the same conditions which here have produced fracture also contribute to rapid wear in service. Dr. P. H. Dudley states:‡ "As I have already observed, the most serviceable rails have a

\* *Journal Iron and Steel Inst.*, 1898, No. 1, p. 178.

† *Journal Iron and Steel Inst.*, 1898, No. 1, p. 185.

‡ *Trans. A. I. M. E.*, 1893, XXIII, p. 651.





fine structure and small mineral aggregates, though in many cases the so-called chemical composition is identical."

In our own work upon bearing metals\* we have invariably found these conditions true, namely, coarse grain and presence of foreign matter result in relatively rapid wear in service.

Turning now to the influence of mill methods upon the size of grain, during the past ten years, marked progress has been made in knowledge regarding the metallurgy of steel, and thanks to the work of efficient investigators, the general relations between structure of steel and its physical properties are so clearly proven that cause and effect in many conditions of mill practice have become matters of definite certainty. The cause of coarse granular form is clearly shown in Prof. Sauveur's valuable papers of 1893 before the American Institute of Mining Engineers,† and of 1899 before the Iron and Steel Institute.‡ Proposition V of the latter states: "The higher the temperature above  $W$  from which the steel is allowed to cool undisturbedly the larger the grains," and Proposition VI, "The slower the cooling from a temperature above  $W$  the larger the grains." In other words, stopping thorough working of the steel while materially above the recalescent point, and allowing to cool slowly, can result only in coarse granular structure, and such structure, other things being the same, has been proved by Professor Sauveur,§ Professor Martens,|| Mr. C. H. Ridsdale,¶ Mr. R. G. Morse,\*\* Dr. Sargent,†† and others, to lower elongation and reduction of area, and to lessen the general toughness of the steel. Professor Roberts-Austen, also, in summing up the results of the important investigation made by the British Government after the St. Neots disaster, concerning loss of strength in rails, states: "The character of the grain, therefore, affords valuable evidence as to

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\* *Jour. Franklin Inst.*, 1900, CXLIX, p. 449.

† *Trans. A. I. M. E.*, XXII, p. 547.

‡ *Jour. Iron and Steel Inst.*, 1899, No. 2, p. 195. *Metallographist*, II, p. 266.

§ *Trans. A. I. M. E.*, 1893, XXII, p. 556.

|| *Mittheilungen aus den königlichen technischen Versuchsanstalten*, XIV, 1896, p. 89.

¶ *Jour. Iron and Steel Inst.*, 1898, No. 1, p. 234; 1899, No. 2, p. 128; 1901, No. 2, p. 75.

\*\* *Trans. A. I. M. E.*, 1899, XXIX, p. 731.

†† *Trans. A. I. M. E.*, Richmond meeting, Feb., 1901.

whether the rail has been rolled at a suitable temperature.... The relative amounts of the pearlite and ferrite, as well as the relative dimensions of the respective granules, afford very valuable indications as to the qualities of the rail and its probable durability.\*

It will thus be seen that the unsatisfactory results which our service tests show with this coarse granular structure are merely the natural accompaniments of such character, and the entire investigation proves clearly the immense practical advantage in the betterment of quality which is being derived from the researches of metallurgists and metallographists, especially during the past decade.

The mills have not been slow to recognize the need of fine-grained structure in the heavier sections, and during recent years great efforts have been made to effect the change, and much progress has followed. At first, attention was directed merely to the finishing temperature; that is, the temperature at which the rail came through the last pass, without particular regard to the initial temperature of the ingot or bloom. In fact, high initial temperatures tended to prevail out of deference to the rolls, especially where these were light, or where the power was limited, and under such conditions it became necessary to hold the rails before running through the last passes, in order to finish at the desired temperature. The result of this procedure, as pointed out by Mr. S. S. Martin recently,† was to produce a fine granular form to the depth to which the working of the steel extended during these last passes, or to a depth of from  $\frac{1}{8}$  to  $\frac{1}{4}$  inch below the surface of the head.

Our own studies had proved the correctness of this statement, and further showed that the size of grain at center of head was but little affected by such treatment, as shown in Fig. 5 (50 diameters). A mere surface toughening resulted, which was of only temporary benefit, and could not from its nature cause any very decided increase in durability in service. The character needed is not a fine-and-coarse irregular structure, but one fairly fine even at the center of head, and practically amorphous at the surface; just such a structure, in fact, as we find in the rails

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\* English Govt. Report on "Loss of Strength in Steel Rails," 1900, p. 58.

† *Iron Age*, Dec. 26, 1901; *The Metallographist*, July, 1902.



of lighter sections rolled before the heavier sections came into use. Fig. 6 (50 diameters) represents the structure at center of head of a 67-pound rail which was rolled and laid in 1864, and which withstood thirty-four years' heavy traffic, and since then has been in use in side track. The analysis is:

Carbon . . . . .	.33	per cent.
Phosphorus . . . . .	.039	" "
Manganese . . . . .	.390	" "
Sulphur . . . . .	.030	" "
Silicon . . . . .	.070	" "

Fig. 7 (reduced 64 per cent) represents the appearance of this rail in 1898, etched, and shows that it had evidently been greatly worn from its original section, and contained a pipe in the web. The metal, however, has not "flowed over" upon the side of the head, although the steel is comparatively soft. In explanation, however, it is to be noted that the head is almost free from foreign matter, and that the structure even at center of head is exceedingly fine.

"Flowing over" has been found by us to be practically independent of the composition, and caused generally by unwelded seams due to presence of foreign matter in the steel. In 1890 Dr. C. B. Dudley stated\* that his studies of such rails "would seem to indicate that disintegration or crushing of steel is largely a resultant of lack of soundness in the ingot, and is more mechanical than chemical, except in so far as chemistry may be responsible for the soundness of the ingot." In some cases, however, we have found almost complete freedom from foreign matter, but in each such instance a coarser granular structure was present. Dr. P. H. Dudley also states:† "The mineral aggregates are large and friable, and the surface of the rail breaks down more than  $\frac{1}{32}$  of an inch in depth, readily flowing under wheel pressures." Thus, it seems clearly established that such condition may arise from marked weakness due to any cause, whether unsoundness of the metal; weak, coarse structure, or even such composition or form that the rail is unable to support the load and becomes flattened.

In the case of the 67-pound rail, Figs. 6 and 7, which gave excellent service, the composition is exceptionally free from

\* *A. I. M. E.*, 1890, XIX, p. 893.

† *Trans. A. I. M. E.*, 1893, XXIII, p. 650.

defects. It is, however, a matter of common experience everywhere that the composition of these old rails of light sections is exceedingly irregular. As Captain Robert W. Hunt expressed it last year before the American Institute of Mining Engineers,\* "they were high in carbon and low in carbon; high in manganese and low in that element; high in phosphorus and higher in phosphorus; but they all gave good service." One such rail taken from our tracks contained .160 per cent phosphorus with .44 per cent carbon, but still had given long service. The one characteristic common to all of these rails is that of fine granular structure; in fact, it was necessitated by the conditions under which they were rolled, for the steel was thoroughly worked well down toward the recalescent point.

In order to note the exact influence of structure upon quality, we took at random from a heat which passed the drop-test a rail having the following composition:

Carbon	.	.	.	.	.	.	.56	per cent.
Phosphorus	.	.	.	.	.	.	.102	" "
Manganese	.	.	.	.	.	.	1.08	" "
Sulphur	.	.	.	.	.	.	.056	" "
Silicon	.	.	.	.	.	.	.147	" "

turned down a section at center of head and obtained the following results:

Tensile strength	.	.	.	.	128,400 lbs. per sq. in.
Elongation in 2 in.	.	.	.	.	8½ per cent.
Reduction of area	.	.	.	.	3½ " "

The structure (50 diameters) at center of head is shown in Fig. 8, and the etched head (reduced 50 per cent) in Fig. 9. The former is very coarse, but the latter is tolerably free from foreign matter.

An adjoining portion of the same rail was then reheated to a cherry red, and let cool at once in the air. A test section was then turned from the center of head, having a structure (50 diameters) represented in Fig. 10, and gave the following results:

Tensile strength	.	.	.	.	129,500 lbs. per sq. in.
Elongation in 2 in.	.	.	.	.	12 per cent.
Reduction of area	.	.	.	.	10.1 " "

\* *Trans. A. I. M. E.*, Richmond meeting, Feb., 1901.



It is thus seen that the change shown in the structure has caused an increase of strength, an increase of nearly fifty per cent in elongation, and an increase of nearly threefold in reduction of area, fully accounting for the marked difference observed in service between the coarser and the finer structures, respectively. As Mr. William R. Webster stated at the Paris Congress of the International Association for Testing Materials, in 1900:\* "The matter of vital importance is to work steel in such manner that it is not put in service with this coarse grain."

In the foregoing we have seen that coarse granular structure seriously weakens steel, and that brittleness under the drop-test is found when in addition the metal is "burned" or contains an excess of foreign matter. The result of a large number of tests further proves that fracture under the drop-test seldom occurs if the steel is homogeneous and free from foreign matter. In other words, mere ability of rails of this composition to withstand the drop-test is no guarantee of fine granular structure, and consequently other tests of quality are essential to necessitate durability. Figs. 1 and 8 represent rails of coarse granular structure which passed the drop-test with good deflection, but such rails do not give satisfactory service.

The foreign matter which produced brittleness was generally found in fine particles or lines scattered throughout the section, but especially prominent within  $\frac{1}{2}$  inch from the surface around the head, and caused a series of unwelded seams—elements of weakness. The appearance was usually similar to that of Fig. 2. Rails which did not fracture under the drop-test at the same rolling were found comparatively free from this enclosed matter around the outer portion of the head, though in some cases a considerable proportion was found nearer the web. Still, the solid band of homogeneous material around the outside of the head gave sufficient toughness to the rail to enable it to resist the force of the drop. It is of interest, however, to note that an excessive proportion of foreign matter in rails may not cause failure under the drop-test in cases where the grain is comparatively fine, since the strength incident to such structure may offset the weakness caused by the oxides. A typical case came under our observation not long ago. A lot of 90-pound rails had been rolled and had passed the drop-test without a

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\* *Engineering*, August 10, 1900, p. 193.

single failure, and the average structure was somewhat finer than that represented in Fig. 3. After being in tracks for a few months a number of the rails began to flow over and break down under the heavy traffic to such an extent that replacement became necessary. Upon inspecting the track we found that some of the rails from the same rolling were in good condition, showing little wear, while others next to them were badly broken down, often along the entire length. The defective rails were not confined to any one heat, but were scattered throughout the rolling. Sometimes the outside edge of a rail was broken down, and sometimes the inside edge. Some in a given heat were showing good service, while one or more from the same heat were defective. Eighty-pound rails of practically the same composition had previously been in this track and had given good service, and had not been broken down by the same traffic.

In order to get at the cause of the difficulty, sections of some of the defective rails were obtained, and showed the following average composition:

Carbon	. . . . .	.544	per cent.
Manganese	. . . . .	1.005	" "
Phosphorus	. . . . .	.078	" "
Sulphur	. . . . .	.090	" "
Silicon	. . . . .	.120	" "

There was nothing abnormal in this, and many rails of essentially the same composition have given good results under heavy traffic conditions.

We next investigated the structure of the steel. Fig. 12 represents the general character of the fracture magnified to  $1\frac{1}{2}$  diameters, and is a longitudinal section down through the head of the rail about  $\frac{1}{2}$  inch from the center line. The striations which appear are unwelded seams which break up the continuity of the steel and produce planes of weakness. In order to learn the extent of these defects we polished off a full section of the rail and etched lightly with iodine, finding that the entire surface of the section was covered with lines of unwelded seams, as represented in Fig. 11 (reduced 52 per cent). Microscopic examination showed that the steel contained a large number of particles of foreign matter — Fig. 13 ( $\times 50$  diameters) — and that some seams were coated with blue and brownish films,



evidence that the steel had been burned,\* and fully accounting for the failure to weld. Fig. 3 gives a section from center of head, and proves that the rail was worked and finished at a moderately low temperature, and that owing to the consequent fairly fine structure the rails withstood the drop-test in spite of the weakness due to defective manufacture. When laid in track, the weight of the rolling stock caused the unwelded seams to slip apart at the portions where the greatest strain came, thus resulting in the breaking down of the rails. The upper right-hand corner of Fig. 11 shows where a sliver  $\frac{1}{2}$  inch thick was forced out of the side of the head along one of the unwelded seams for a distance of several feet, and the crack along the seam extended nearly to the top of the head.

This instance proved conclusively that reliance could not be placed upon the drop-test to ensure homogeneous, solid steel, and it is generally well known that rails of laminated steel may give even a better test under the drop than if composed of solid metal of the same composition.

The above results clearly verify the statement of Dr. P. H. Dudley† that solidity and continuity are quite as important as a good microstructure.

As to the origin of foreign matter in steel, it is evident that it must be due to defective mill practice either in the manufacture of the steel, including turning and settling; in burning of steel in soaking pits — or in blooming furnaces, if the latter are used — or in cropping blooms or rails; in any event it is an unquestioned proof of defective mill practice, and is easily avoided if so desired.

In order to ensure the most durable rail of a given composition, our service results, as shown above, indicate that there must be —

- (1) Freedom from brittleness.
- (2) Absence of foreign matter in undue proportion.
- (3) Fine granular structure.

(1) Brittle rails are eliminated by the drop-test of 2,000 pounds falling 20 feet upon a rail-butt from the top of the ingot, one rail from each heat being tested, or, in case of fracture, two more being taken from the tops of other ingots of the same

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\* J. E. Stead: *Journal Iron and Steel Institute*, 1898, No. 1, p. 183.

† *Trans. A. I. M. E.*, 1893, XXIII, p. 652.

heat; fracture of two out of the three causing rejection of all rails in the heat.

(2) Absence of foreign matter is best ensured by careful inspection throughout the manufacture, and by specifying, a definite proportion of cropping.

(3) Fine granular structure is necessitated by specifying under definite conditions the shrinkage of the rail after leaving hot saws — an idea suggested by Mr. Wm. R. Webster.\*

A shrinkage limit is in operation in most of the mills to-day, but it must be borne in mind that, as pointed out by Mr. Martin in the paper above referred to,† that specifying merely the distance between hot saws does not compel presence of fine grain clear to center of head. Such structure will not result unless the reduction in the passes after the holding is sufficient to work the steel thoroughly to the center.

What is needed is a moderately low initial temperature, sufficient work to render the steel solid, the speed of train and extent of reduction being such that with rapid rolling — without undue holding before or in the last passes, and without artificial cooling subsequently — the distance between hot saws shall not exceed a specified amount.

Under the above conditions, with a given composition, the extent of shrinkage of the rail after leaving hot saws may be made a definite guarantee of size of grain in sections of a given weight, and if the limit is placed at  $5\frac{1}{2}$  inches for a 30-foot length of a 90-pound rail, coarse grain even at center of head is practically impossible.

It is, of course, obvious that mere fine grain does not render freedom from brittleness certain, as, for instance, in the case of the St. Neots rail above referred to,‡ where the steel was fairly fine-grained, but was fragile owing to presence of martensite due to rapid cooling from a high temperature. Such composition was, however, evidently caused by a very exceptional accident in mill practice, and is carefully guarded against under normal conditions.

In working toward the most efficient practice in rail roll-

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\* *Trans. Am. Soc. C. E.*, XLIV, p. 497.

† *Iron Age*, Dec. 26, 1901.

‡ Page 182.



ing, valuable aid has been given during the past few years by Committee No. 1 of the American Section of the International Association for Testing Materials, and Mr. Wm. R. Webster, the chairman, deservedly receives great credit for his efforts in bringing together the various conflicting interests, and in formulating proposed standard specifications of various materials of construction. The specifications for steel rails in particular have aroused wide discussion. In its present form we regard it as tentative, since it merely specifies the quality which, since the introduction of the heavier sections, has everywhere been found rapid wearing and unsatisfactory. It does not ensure durable material, and it does not represent the best American practice. It will, however, serve as a basis to which the necessary additions or modifications can be made, and also its effect in provoking discussion will be of assistance. If so drawn as to represent the best American practice, it would benefit the railroads as a class, and would undoubtedly be of great commercial value to the mills as an influence upon foreign orders, and would go far toward proving that many rail failures and cases of brittleness attributed abroad to chemical composition have been simply results there of defective mill practice which would not be found under the given best American practice. As an instance, we may cite the statement of Mr. C. P. Sandberg before the International Railway Congress in 1901,\* that he made some experimental tests with rails containing up to .60 per cent carbon, and met with complete failure. The rails were brittle, and broke under a drop-test of 1 ton falling 10 feet. The carbon ranged from .56 per cent to .62 per cent. In contrast to this we have the regular practice of our American mills, where, as shown above, failure under the drop-test of 2000 pounds falling 20 feet seldom occurs, even with carbon considerably beyond the .60 per cent limit, provided proper care is exercised in manufacture.

The specifications proposed to the American Railway Engineering and Maintenance of Way Association† are an improvement over those of the International Association in inserting a shrinkage clause, but the wording of the latter is open to

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\* *Bulletin International Railway Congress*, English Ed., XV, p. 1647, July, 1901. Also *Jour. Iron and Steel Inst.*, 1898, No. 2, p. 78.

† *Amer. Railway Engineering and Maintenance of Way Asso. Bulletin*, 22, March, 1902.

the objection cited by Mr. Martin,\* and would ensure nothing more than fine-grained material upon the immediate outer surface of the rail.

Every successful specification is of necessity based directly upon the teachings of actual service. In order, then, to aid the mills in working out the most efficient standard practice, it is greatly to the interest of every consumer to make known the practical service values of the various materials. Our object in entering upon this investigation has been, naturally, to secure the greatest degree of safety and durability in the tracks of the Reading Railway, but we have brought the results to your attention in the hope that a full and free discussion of facts may be of general benefit in tending toward the production of uniformly durable material.

In conclusion, I wish to thank my assistants for the analytical and inspection work carried on in connection with this investigation.

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## RAIL ROLLING AT LOWER TEMPERATURES DURING 1901 †

By S. S. MARTIN

FOR years it has been known that light rails have a very much more non-granular structure than the heavy sections, which is caused by their being finished at a lower temperature. This structure means to the railroad people a better wearing rail. The latter have never developed the reason for it. To people asking the question, "Why did our rails rolled years back wear better?" we can easily answer that since the rails were lighter and since the methods were not improved for great tonnages, a structure was obtained which was nearer to the non-granular. It has only been in the last year that the subject has taken a practical turn in the direction of rolling the heavier sections (say 70 pounds and up) nearer the temperature of the lighter

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\* Above, pages 8 and 15.

† *The Iron Age*, December 26, 1901. This article and the illustrations are reproduced here through the courtesy of *The Iron Age*.



rails. When the railroads made their rails heavier the manufacturers did not meet promptly the necessity of rolling their heavy rails at a lower temperature. Now the manufacturers find themselves pushed to do this, with the result that some of the mills reheating their blooms find that they must adopt some means of cooling the bar somewhere about the rail rolls, so as to roll at a very much lower temperature. To appreciate fully what low temperature for rolling may really mean, I will briefly speak of rolling temperatures.

When steel cools from the rolling or finishing temperature there are certain points at which for an instant there is a retardation in the falling of the temperature, and sometimes a perceptible increase of heat. Soft steel, say under .20 per cent carbon, has three of these points, while rail steel of .50 per cent carbon has only one. These points are called the "points of recalescence," or, commonly, the "critical points." The structure of steel does not change below this point to any noticeable extent. Steel with .20 per cent carbon or under has three of these points, at 650, 740 and 860° C., while rail steel of, say, .45 to .55 per cent carbon has only one critical point, which lies (according to different writers) between 700 and 725° C. The structure of the rail depends for the size of its granules on the rate of cooling to the critical point, and the higher the temperature above the critical point the larger will be the grains. Since no change of structure can result from finishing below the critical point, we naturally decide that a rail must be rolled as near the critical point as possible to get the best structure, or in the case of rail steel between 700 and 725° C. If the piece of steel in rolling is delayed at any point before finishing it will help the structure directly in proportion to the amount of reduction in the remaining passes, because while the bar is being held (the temperature when starting to hold being about 950 to 1000° C.) the structure is crystallizing, and the higher the temperature the larger the granules to the critical point. For this reason the amount of reduction greatly influences the granular structure, and it should be considerable in order to move the grains to any depth in the bar. The result generally obtained from a number of microphotographs of this class of finished rails is that the steel is non-granular just as far as the reduction in the last pass thoroughly moves the mass, which for a 5 per cent reduction will be from

$\frac{1}{8}$  to  $\frac{1}{4}$  inch; the rest of the distance to the center of the head becomes abruptly more granular from the  $\frac{1}{8}$  to  $\frac{1}{4}$  inch until the center of the head, which shows the largest granules.

It thus seems that to obtain a non-granular structure throughout the rail the temperature must be reduced earlier in the rolling — in fact, the bar or bloom must not be reheated, but should be rolled direct from the ingot. In the latter case the bloom will be delivered to the rail rolls at a temperature of  $850^{\circ}$  C., while a reheated bloom will be delivered at  $1000^{\circ}$  C., or more. I want to emphasize the fact that while the rail is held before the final pass, at a temperature above the critical point, it crystallizes, and the single pass which it subsequently receives is incapable of breaking up the granular structure for more than a certain depth. This practice of direct rolling has been carried on for years in some mills in this country, and in almost all mills abroad. There is no trouble with a granular structure, while the mills rolling reheated blooms have run hard against very granular structure, and thus have this year introduced the method of holding the bar before the last pass. This has greatly improved their structure, but it is not as homogeneous as a structure obtained by direct rolling, as the temperatures are so much above the critical point that the steel becomes granular so easily while being held for the cooling before the final pass.

The microscopic study of rails from these different methods of rolling is interesting, and plainly shows that the bar must be kept at a lower general or average temperature to get the best structure throughout. Usually the method of holding for one and three-quarters to two minutes at  $900$  to  $1000^{\circ}$  C. before the bar goes through the finishing pass allows a very granular structure to develop, which the reduction of the last pass of 5 per cent will not destroy to a greater depth than  $\frac{1}{8}$  to  $\frac{1}{4}$  inch toward the center of the head, which, as far as temperature is concerned, is far enough above the critical point to do some more crystallizing after the finishing. If not, the granular structure found comes from the first cause explained — i.e., a per cent of reduction too small to reach the center effectively.

Our microscopists publish occasionally micro-photographs showing the structure obtained by forging at certain known temperatures, measured with the Le Chatelier pyrometer. Professor



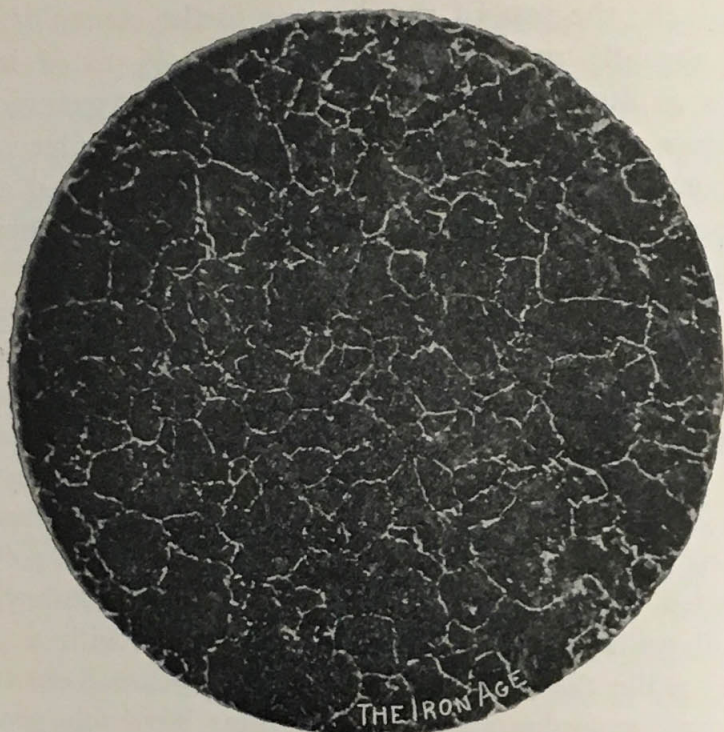


Fig. 1. Center of head.

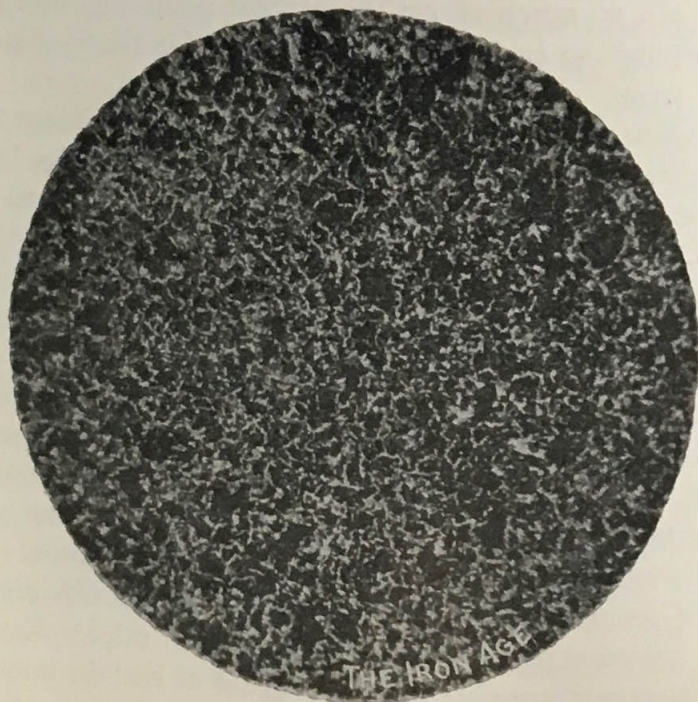


Fig. 2.  $\frac{1}{8}$  inch from surface of head.

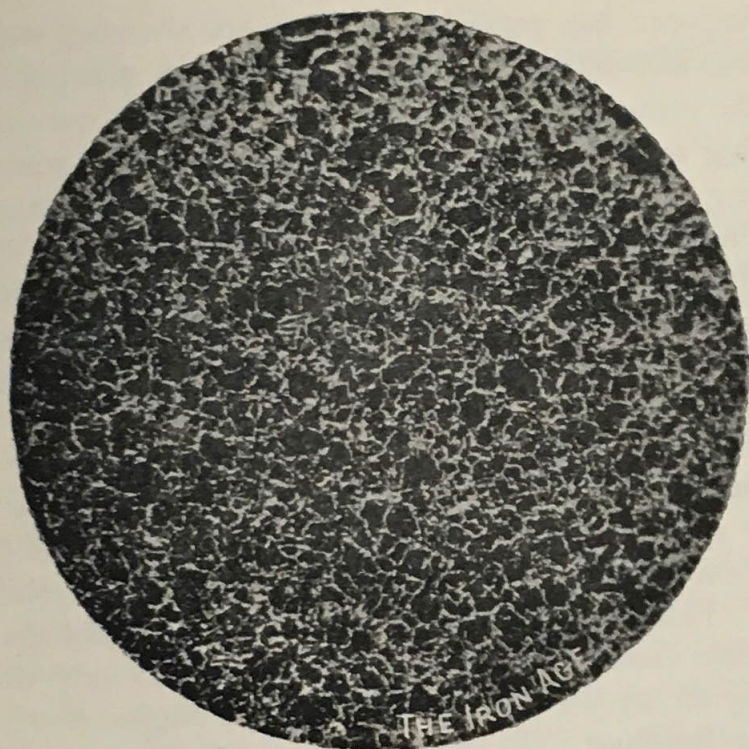


Fig. 3. Center of head.

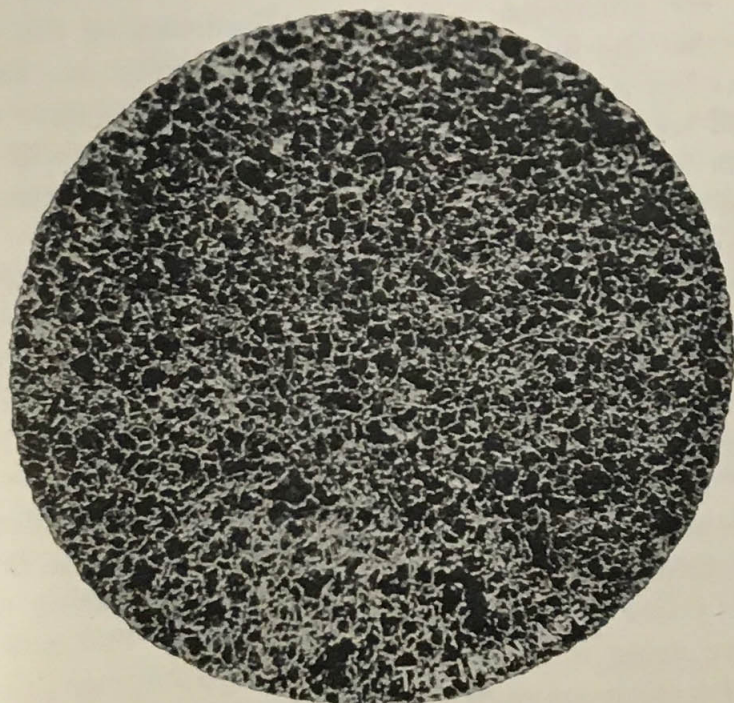


Fig. 4.  $\frac{1}{8}$  inch from surface of head.



Albert Sauveur has published such a set showing steel forged at 650, 850 and 1100° C., which, if used as a standard for comparing the structures obtained in rails, gives at once a method of knowing actually what temperature the rail had when cooling after being finished and just how far above the critical point. Without a doubt, the proper way to get at this matter of rolling temperatures is through the microscope. This idea is rapidly gaining ground, and the microscopists deserve great credit for the manner in which they have brought forward the microscope practicably.

I am rather disappointed at the method which the railroad people use to decide what the temperature has been by putting into their specifications a shrinkage clause, in the belief that this will insure a non-granular structure and hence a low rolling temperature. This is not the correct idea, because shrinkage of different steels varies. Then, too, it is so easy to vary shrinkage by holding at the last pass, by running the engine slowly which drives the finishing rolls, and by the application of water both before (which will help the structure) and after (which does not generally have much effect, as steel is near the critical point). Now a non-granular structure of the rail is ideal, although not practicable, but the requirements of the railroads are such that the mills are all striving to outdo one another in getting as low a temperature as possible and also have the head of the rail homogeneous. Microscopic examination will give the true result, something that cannot be influenced by mankind, as shrinkage can.

In conclusion I desire to remind my readers that shrinkage continues under the critical point, and hence does not in the least help or indicate the structure. So it seems that shrinkage is very misleading to the railroad people.

The following micro-photographs explain the above article and were obtained in the following manner:

Figs. 1 and 2 are from the first half of an ingot rolled as hot as possible through the ten passes of the rail rolls to the final pass and there held  $1\frac{3}{4}$  minutes; then through the finishing pass.

Figs. 3 and 4 are from the other half of the same ingot, held before going into the rail rolls for two minutes; then sent through the 11 passes at this reduced temperature.

## STRUCTURE AND FINISHING TEMPERATURE OF STEEL RAILS\*

By ALBERT SAUVEUR

IT is well known that when a piece of steel is allowed to cool undisturbedly from a high temperature, it crystallizes, and that the resulting crystals or grains, as they are frequently called, are the larger, the higher the initial temperature and the slower the cooling. This was first forcibly stated by Professor Tschernoff in his masterly paper on "The Manufacture of Steel and the Mode of Working it," communicated to the Russian Technical Society in April and May, 1868.

It is also well known that if the steel be vigorously worked (rolled or forged) while it is cooling from a high temperature, crystallization is prevented, but as soon as work ceases, crystallization sets in until a certain temperature is reached, which in the majority of cases is not far from  $700^{\circ}$  C., and below which there is no further growth of crystals. If work be performed below that critical temperature the crystals are permanently distorted, being flattened and elongated in the direction of the rolling, and this distortion of the crystals is greater the lower the temperature at which the work is carried on. Such distortion causes a decrease of ductility and, eventually, brittleness, a striking illustration of which is found in the drawing of wire, which must be frequently annealed to remove the brittleness produced by work at a low temperature. The annealing removes the distortion of the crystals and, therefore, the resulting brittleness by causing the metal to assume a new and finer crystallization.

We may, for convenience, call the range of temperature during which steel crystallizes, and which extends from the melting point to the critical temperature (about  $700^{\circ}$  C. in the case of carbon steel), the crystallizing range, and the work performed at these temperatures *hot work*, while the work done below these temperatures will be called *cold work*.

Hot work influences the final structure by preventing or retarding crystallization, while cold work distorts the crystals.

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\* Read at the Fifth Annual Meeting of the American Section of the International Association for Testing Materials, Atlantic City, N. J., June 13, 1902.



It follows from the above considerations that in working steel, as is done in the manufacture of so many implements, from a high to a much lower temperature, crystallization is retarded, i.e., is made up to cover a much shorter range of temperature, extending from the *finishing temperature* (i.e., the temperature at which work ceases) to the critical temperature. The resulting structure will, therefore, be finer grained, i.e., will be made up of smaller crystals, than if the metal had been allowed to cool *undisturbedly* from a high temperature: the crystals will be the smaller the lower the finishing temperature. Should work be continued below the critical temperature the crystals will be distorted, which implies a loss of ductility.

It has been conclusively shown that the finer the structure, i.e., the smaller the crystals, the more ductile will be the steel, and since ductility is always a very desirable property, whatever the intended use of the finished implement, we should so conduct our treatment of the metal as to confer upon it the finest possible structure. The importance of finishing steel implements at the proper temperature, therefore, need not be insisted upon. It is now appreciated by all enlightened metallurgists.

The manufacturers of steel rails have, more than any other producers of finished steel articles, given careful attention to the important influence of the finishing temperature upon the structure and the physical properties of their rails. In these days when the tendency is to allow more and more carbon in rail steel, in order to lengthen the life of the rail, the importance of securing all the ductility possible from the heat treatment stands preëminently at the front.

The importance of low finishing temperatures was clearly brought out some ten years ago at the South Chicago works of the Illinois Steel Company, through some extensive tests conducted by the writer, the results of which were summed up in a paper presented at the Engineering Congress in 1893. In spite of the conclusiveness of these tests, however, the management could not be made to take up any efficient measure toward lowering the generally too high finishing temperature of the rails. It was assumed that any step in that direction would interfere with the output and this was not to be considered. While the results given in the paper did not attract much attention at the time, a seed had been sown that was destined to bear much fruit. The

importance of proper finishing temperatures gradually asserted itself until at the present day there is hardly a steel rail user or producer who is not fully alive to it. The results of many experiments along this line have been published within the last twelve months, which, although they merely confirm the conclusions reached some ten years ago are, curiously enough, treated by their authors as newly discovered facts. This peculiar attitude on the part of writers who ought to be familiar with the previous treatment of their subject, attracted the attention of the *Railroad Gazette*, which says editorially, under date of May 31, 1901:

In all the discussions on the heat treatment of steel the matter has been treated as though it were new, and the writers appear to have forgotten the early work of Tschernoff, Metcalf and others, and the later work in the early 90's. It is well to realize thoroughly that this is no new fad. Notwithstanding all that has been said on this subject it has been a hard matter to get engineers to realize that steel can be rendered almost worthless by being finished too hot in rolling. When this important fact is fully appreciated we may expect to get the best results from steel, as means will be taken to give it the proper heat treatment in rolling.

In Mr. Metcalf's classical paper on steel, presented before the American Society of Civil Engineers, March 2, 1887, and reprinted in the *Railroad Gazette* of March 18, he says: "As steel congeals it forms crystals and the sizes are largely affected by the rate of cooling; slow cooling favoring the formation of large crystals, quick cooling and also agitation producing fine crystals... In every piece of steel that is in existence to-day there is a sure record of the last temperature to which it was subjected as well as of the manner in which the steel was worked. I mean to say that for every variation of heat that is visible to the naked eye there is a corresponding variation in structure which is equally visible to the naked eye if the record be opened by fracturing the piece... For a double quantity of carbon we have five times the difference in specific gravity, due to an equal difference in temperature... The finest crystals and the best structure can only be formed by quick cooling and the violent agitation of the hammer or of the rolls."

At the World's Fair Congress of Engineers, in Chicago, Mr. Albert Sauveur, then in the employ of the Illinois Steel Company, gave the results of his investigation on the heat treatment of steel rails in six propositions, which are well worth the study of any one interested in this subject. His paper will be found in the *Transactions of the Institute of Mining Engineers*, Vol. XXII, from which we quote:

"A polished and etched section of steel rail when examined under the microscope does not by any means reveal in all its parts the same structure. This heterogeneousness of structure is due to the different temperatures at which the various parts of the rail leave the finishing-



rolls, and to the unequal rate of their subsequent cooling. The propositions formulated in the first part of this paper should enable us to foretell such variations in the structure of a rail.

"The outside of a rail, leaving the rolls coldest and cooling quickest, will offer less chance for crystallization, and should therefore show a smaller grain than the inside, which, being the hottest and cooling down more slowly, will favor the crystallization of the metal, the size of the crystals or grains reaching its maximum in the centre of the head. It might also be inferred that the smallest grain will be found at the extremities of the flange, since this is always the coldest-finished region. Proceeding from the outside to the inside we shall find the tendency toward coarse crystallization gradually increasing."

*The Kennedy-Morrison Rail Finishing Process.* — The suddenly renewed interest in the subject of proper finishing temperature for steel rails, just alluded to, led the Carnegie Steel Company to a notable departure at their Edgar Thompson plant, from the ordinary mode of proceeding. This move on their part was given much publicity and has attracted the attention of all rail consumers. Stated briefly, the modification which they have introduced in the rolling of steel rails consists in holding the rails on a cooling table a few minutes before subjecting them to the last pass. It is argued that by so doing the rail is finished at a lower temperature, which is the desideratum aimed at.

Let us look critically into this new departure in rail rolling. The rail when it reaches the cooling table introduced before the final pass is at a high, or relatively high, temperature and is then allowed to cool until a certain lower temperature is attained. During this undisturbed cooling the metal evidently crystallizes, and the rail must necessarily enter the finishing rolls in a crystalline condition, which will be the more pronounced the higher the temperature of the rail when it reached the cooling table and the slower and more prolonged the subsequent *undisturbed* cooling. It also seems as if the single pass to which the metal is subjected after this crystallizing period cannot be sufficient to break up the crystallization just created. On theoretical grounds, therefore, if a material gain results from this method of proceeding, I do not see how it can be accounted for. Let us remember that any *undisturbed* cooling above the critical temperature constitutes a crystallizing period. By partially cooling the rail before the final pass, we shorten the crystallizing range after the rail has left the finishing rolls, but we create a crystallizing period just before the

final pass, which is exactly equal in length to the diminution of the final undisturbed cooling. We do not, by so doing, decrease the *total* crystallizing range, which alone governs the size of the final crystals; we merely divide it, causing a part of it to be located immediately before the final pass and the balance immediately after, so that a rail treated by this process has passed through a crystallizing period of *exactly the same length*.

The only beneficial effect, therefore, that can be claimed for this process is that it breaks up the crystals produced just before the last pass, resulting in a finer structure rail. If we consider that the reduction produced by the last pass is about five per cent it seems hardly conceivable that so slight an amount of work could be effective in breaking a preëxisting coarse structure.

Similar criticisms of the value of the Kennedy-Morrison process have been published by Mr. S. S. Martin, of the Maryland Steel Company, who contends, apparently on good ground, that any undisturbed cooling introduced in the process of rolling, in order to be effective in producing a finer grained rail, must take place at an early period, because then the considerable reduction, i.e., the great amount of work to which the rail is subsequently subjected, will break up the crystals formed during that undisturbed cooling. It has been also argued, and rightly, I think, that those mills which do not reheat their blooms produce finer grained rails, for the simple reason that they finish their rails at a lower temperature.

To sum up, in order to confer a fine-grained structure upon steel rails, three courses seem to be opened:

1. To shorten the crystallizing period, i.e., the time during which the rail is allowed to cool undisturbedly above the critical temperature (say about  $700^{\circ}$  C.). In other words, the rail should be finished sufficiently cooled, and the finishing temperature will, of course, depend in turn upon the temperature of the ingot when rolling begins and upon the speed of the operations. The main objection to starting and carrying the rolling at a relatively low temperature is that it throws more work upon the rolls. The objection to a slow rolling speed and handling of the material is that it decreases the output of the mill. Both objections imply an increase in the cost of the rail.

2. To cause a part of the crystallizing period to occur previous to the final pass, i.e., to allow the rail to cool undisturbedly



at some stage of the operations, and for a short time, before completing the rolling. This cooling in order to be effective should occur at a sufficiently early stage of the rolling, in order that the resulting crystallization may be broken up through a sufficient amount of subsequent work and reduction. Such results would be obtained by placing the cooling table at the shears. The objection to this course is that the shaping of this cooler bloom into rails would require greater mechanical effort and, therefore, increase the cost of production.

3. To finish the rail at the temperature most desirable for easiness and speed of manipulation in rolling and then to reheat it to a temperature slightly above the critical temperature, a treatment which would result in the breaking up of the preëxisting coarse structure, replacing it by a much finer one. This course would undoubtedly be the most effective of the three, as it would not only impart a fine structure to the rail, but also a *uniform* structure to all its parts, while a rail not so treated must necessarily be coarser in the central parts than nearer the cooling surfaces, owing to the higher temperature of the centre when the rail leaves the finishing rolls and to its lower subsequent cooling. The cost of such reheating treatment would undoubtedly be very great and, possibly, prohibitive.

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## THE EQUILIBRIUM OF IRON-CARBON SYSTEMS\*

By G. CHARPY and L. GRENET

BAKHUIS ROOZEBOOM in 1900 gave an interpretation of the facts known with regard to the constitution of metals formed of iron and carbon, which is irreproachable from a theoretical point of view, but some points of which are doubtful in consequence of the insufficiency of actual exact experiment. In particular, the conditions under which Mr. Roozeboom holds that the carbon separates in the form of graphite in white iron submitted to reheating are evidently in contradiction with a certain

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\* *Bulletin de la Société d'Encouragement*, March, 1902. Translated in *American Manufacturer*, June 5, 1902.

number of facts previously observed in the manufacture of malleable castings. Messrs. Le. Chatelier and Stansfield have objected to this point of view, and quoted the experiments of Royston, Mannesman, etc., according to which the separation of graphite would follow a course distinctly different to that which has been adopted by Mr. Roozeboom. In these experiments, carried out from a practical point of view, substances other than carbon contained in the castings have not been taken into account, although certain of these substances, such as silicon, manganese, etc., possess an incontestable influence over the separation of graphite. It can be believed, as Hugh P. Tiemann points out in a work recently published,\* that the abundant separation of graphite, observed by Mr. Royston, is due to the presence of silicon in the irons used.

We have studied a great number of examples from this point of view, and give in particular the results obtained from five samples containing practically the same amount of total carbon, in which the other elements were in small quantities, except silicon, which existed in variable amounts.

The following table shows the composition of these irons:

	Carbon	Silicon	Manganese	Sulphur	Phosphorus
No. 1	3.60	0.07	0.03	0.01	traces
No. 2	3.40	0.27	traces	0.02	0.02
No. 3	3.25	0.80	traces	0.02	0.03
No. 4	3.20	1.25	0.12	0.01	0.01
No. 5	3.30	2.10	0.12	0.02	0.01

These metals had been cooled in cold water, and did not contain appreciable quantities of graphite, except the last, which contained 0.20 per cent.

Fragments of these irons were submitted to more or less prolonged re-heatings at different temperatures. These temperatures were obtained by heating up and then cooling after a high enough temperature had been reached; in each case the operation was ended by quenching in cold water. In the samples thus treated the total carbon and the graphitic carbon were determined, the difference giving the combined carbon.

The graphite was estimated, following Ledebur's process, by dissolving the metal in 1.18 nitric acid, and burning in oxygen.

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\* *The Metallgraphist*, Vol. IV (1901), p. 313.



the residue remaining insoluble after an hour's evolution. It is, perhaps, well for us to specify that what is called graphite in this paper is the residual carbon insoluble in nitric acid, as is usual in metallurgical research. The amount of silicon was also determined in each sample. From these results we can deduct the following conclusions relating to the separation of graphitic carbon by reheating. We give in each example the observed amount for graphite and combined carbon, though the sum is not exactly constant for each sample. It is necessary then to reckon the value of these two amounts in order to eliminate the influence of variations of carbon due to a partial decarburization during reheating, or to irregularities in composition of an ingot, which we have not thought necessary to correct.

1. *The separation of graphite begins at a temperature which is the lower, the greater the percentage of silicon.*

Thus, with sample No. 1, which contains only traces of silicon, a prolonged heating at 1100 degrees or lower does not give place to any separation of graphite. But at 1150 degrees the separation takes place.

In No. 2, after heating for four hours at 700 degrees, 800 degrees, 900 degrees, and 1000 degrees, not a trace of graphite is to be seen. But it appears after heating to 1100 degrees.

In sample No. 3 traces appeared after heating to 800 degrees.

In samples Nos. 4 and 5 it was sufficient to heat to only 650 degrees to separate the carbon. In the latter, particularly after heating for six hours at 650 degrees, the percentage of graphite rose from 0.10 per cent to 2.83 per cent.

2. *The separation of graphite, once begun, continues at temperatures lower than those at which it begins.* Thus a piece of No. 1 iron heated to 1170 degrees, and quenched, contained only 0.50 per cent graphite and 2.61 per cent combined carbon. Another piece of the same metal heated at the same time to 1170 degrees, and cooled slowly to 700 degrees, and then quenched, contained 1.87 per cent graphite and 0.43 per cent combined carbon. Similarly, a piece of No. 3 iron heated to 1170 degrees, and quenched, contained 1.42 per cent graphite and 1.69 per cent combined carbon. Another piece heated to 1170 degrees and cooled slowly to 700 degrees, and then quenched, contained 2.56 per cent graphite and 0.38 per cent combined carbon.

3. *At constant temperature the separation of graphite is*

effected progressively more feebly as the temperature is lower and the amount of silicon is less.

This is shown by the following results, in which the first column gives the temperature, the second the graphitic carbon, and the third the combined carbon.

No. 3 Iron, 0.80 per cent Silicon				No. 4 Iron, 1.20 per cent Silicon				No. 5 Iron, 2 per cent Silicon			
Hours				Hours				Hours			
One	800	0.10	3.19	One	700	0.06	3.42	One	700	1.39	1.90
Four	800	0.22	3.07	Two	700	0.11	3.30	Two	700	2.09	1.19
One	900	0.30	2.97	Four	700	0.20	3.13	Four	700	2.67	0.28
Two	900	0.60	2.40	One	800	0.12	3.08	One	800	2.36	0.78
Four	900	1.58	1.14	Two	800	0.51	2.47	Two	800	2.31	0.89
One	1,000	0.37	2.94	Four	800	1.64	1.56	Four	800	2.43	0.54
Two	1,000	1.50	1.41	One	900	2.28	0.90	One	900	2.33	0.88
Four	1,000	1.47	1.29	Two	900	2.32	0.90	Two	900	2.32	0.90
				Four	900	3.35	0.99	Four	900	2.33	0.90

It is seen that in the case of No. 5 iron the equilibrium is obtained at the end of one hour at 900 degrees, since the amount of graphite does not vary when the heating is continued for four hours. In No. 3 iron, with 0.80 per cent silicon, it is far from being attained after four hours' heating at the same temperature. In the irons poorest in silicon, the phenomenon is not seen, because the transformation does not occur at temperatures less than 1100 degrees in spite of prolonged heating.

4. *The amount of combined carbon which corresponds to the equilibrium at a given temperature diminishes when the amount of silicon increases.*

The following table shows the figures found after four hours' heating at different temperatures. For the samples Nos. 1 and 2 the reaction was started by heating to 1150 and 1100 and cooling slowly to the proper temperature:

Samples	To 1100 deg.		To 1000 deg.		To 900 deg.		To 700 deg.	
	Graphite	Combined Carbon	Graphite	Combined Carbon	Graphite	Combined Carbon	Graphite	Combined Carbon
No. 1	1.15	1.74	1.03	1.74			1.87	0.43
No. 2	1.26	1.93	1.00	1.62				
No. 3	1.61	1.26	1.60	1.52	1.67	1.17	2.56	0.38
No. 4	2.10	1.02	2.20	0.98	2.32	0.90		
No. 5	2.18	1.00	2.10	0.93	2.33	0.90	2.67	0.28



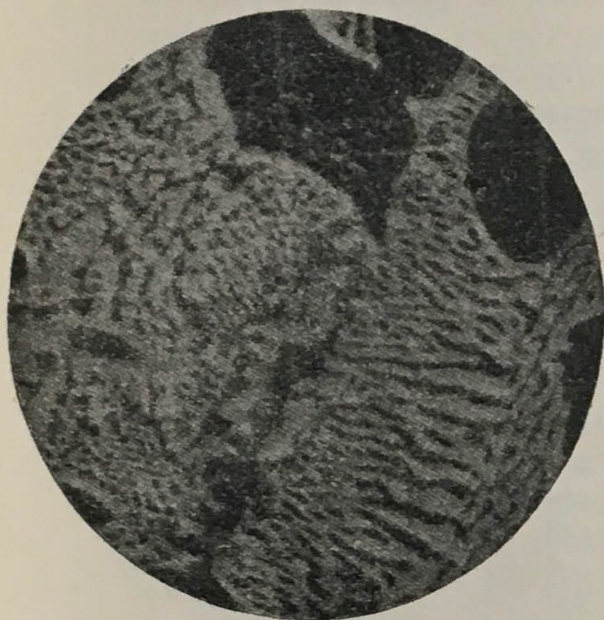


Fig. 1. White cast iron.

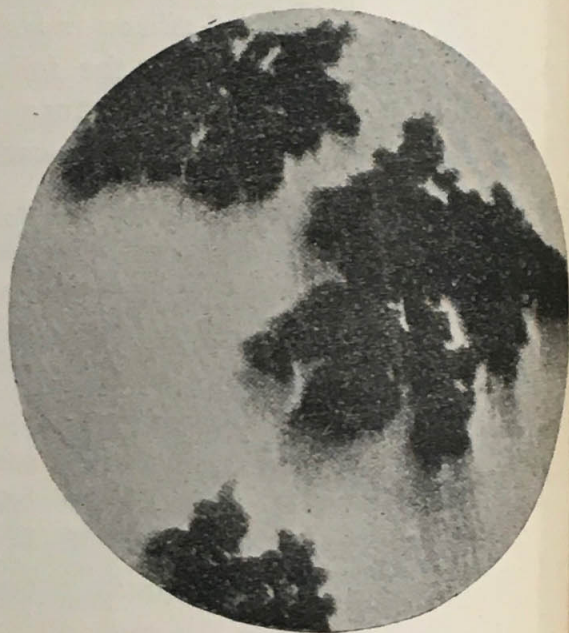


Fig. 2. Cast iron No. 2. Reheated to  $1000^{\circ}\text{C}$ .  
Polished but not etched.

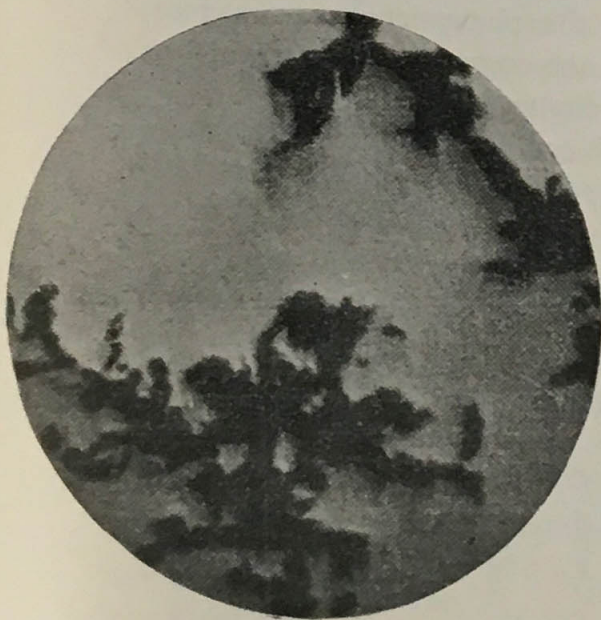


Fig. 3. Cast iron No. 3. Reheated to  $1000^{\circ}\text{C}$ .  
Polished but not etched.

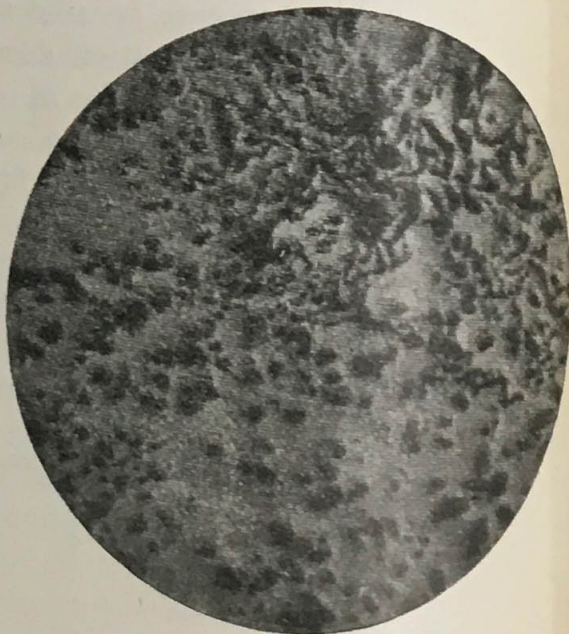


Fig. 4. Cast iron No. 5. Reheated to  $1000^{\circ}\text{C}$ .  
Polished but not etched.



5. *The amount of combined carbon which corresponds to the equilibrium diminishes as the temperature decreases.*

The figures given above already indicate this diminution. We give, however, the results obtained in two series of experiments, in which we have tried to approach, so far as possible, the state of equilibrium. For this purpose, samples of 1 and 3 irons, surrounded by pulverized charcoal, have been heated to 1170 degrees, and then cooled very slowly; at a rate of about 50 degrees per hour, from different temperatures; maintaining these temperatures for two hours and then quenching. For temperatures below 900 degrees, operations lasting more than a day were interrupted and continued the next day; that is to say, after reaching 900 degrees, the sample was allowed to cool, and the following day reheated to 900 degrees, and the slow cooling of the samples continued. The following results were thus obtained:

Temperatures deg.	Sample No. 1		Sample No. 3	
	Graphite	Combined Carbon	Graphite	Combined Carbon
1170	0.50	2.61	1.42	1.69
1100	1.15	1.74	1.49	1.48
1000	1.03	1.74	1.35	1.55
900			1.91	0.99
800	1.15	1.31	2.09	0.43
700	1.87	0.43	2.56	0.38

The microscopic examination of the different samples treated confirms the results of chemical analysis, without adding much information. In the primitive state the samples all present the well known aspect of white irons (Fig. 1). When irons free from silicon are reheated to different temperatures, the respective dimensions of the constituents are modified; and the concentration of the martensite varies according to laws already known; in the silicious irons the graphite is easily visible by simple polishing without chemical attack. The nature of the separated graphite varies with the temperature of separation and with the amount of silicon.

When we examine certain samples in which it has been tried to obtain the equilibrium at the lower temperatures, we notice that the transformation is more advanced in certain regions where the graphite is found in direct contact with the ferrite (Fig. 6).



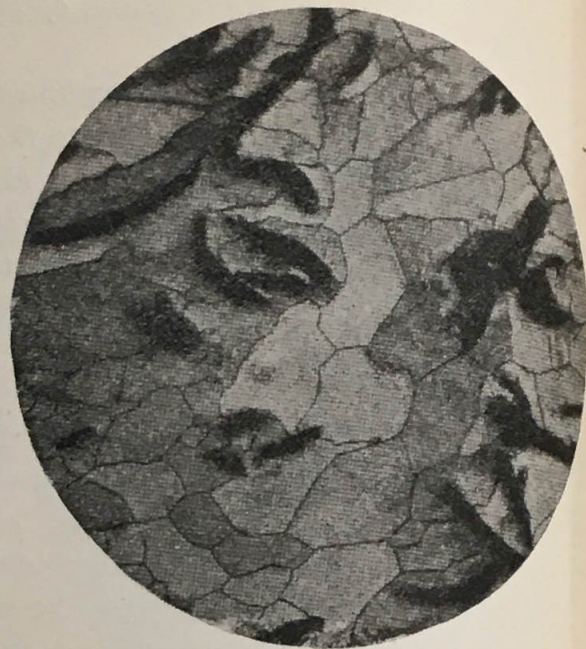
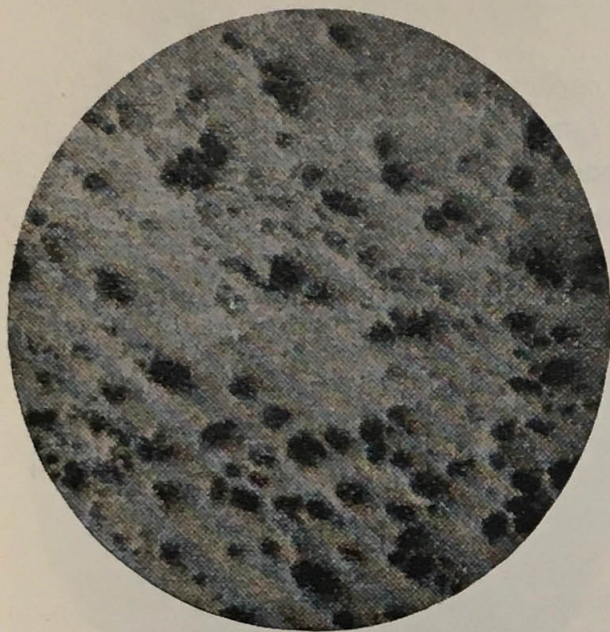


Fig. 5. Cast iron No. 6. Reheated to 800° C.  
Polished but not etched.

Fig. 6. Ferrite and graphite.

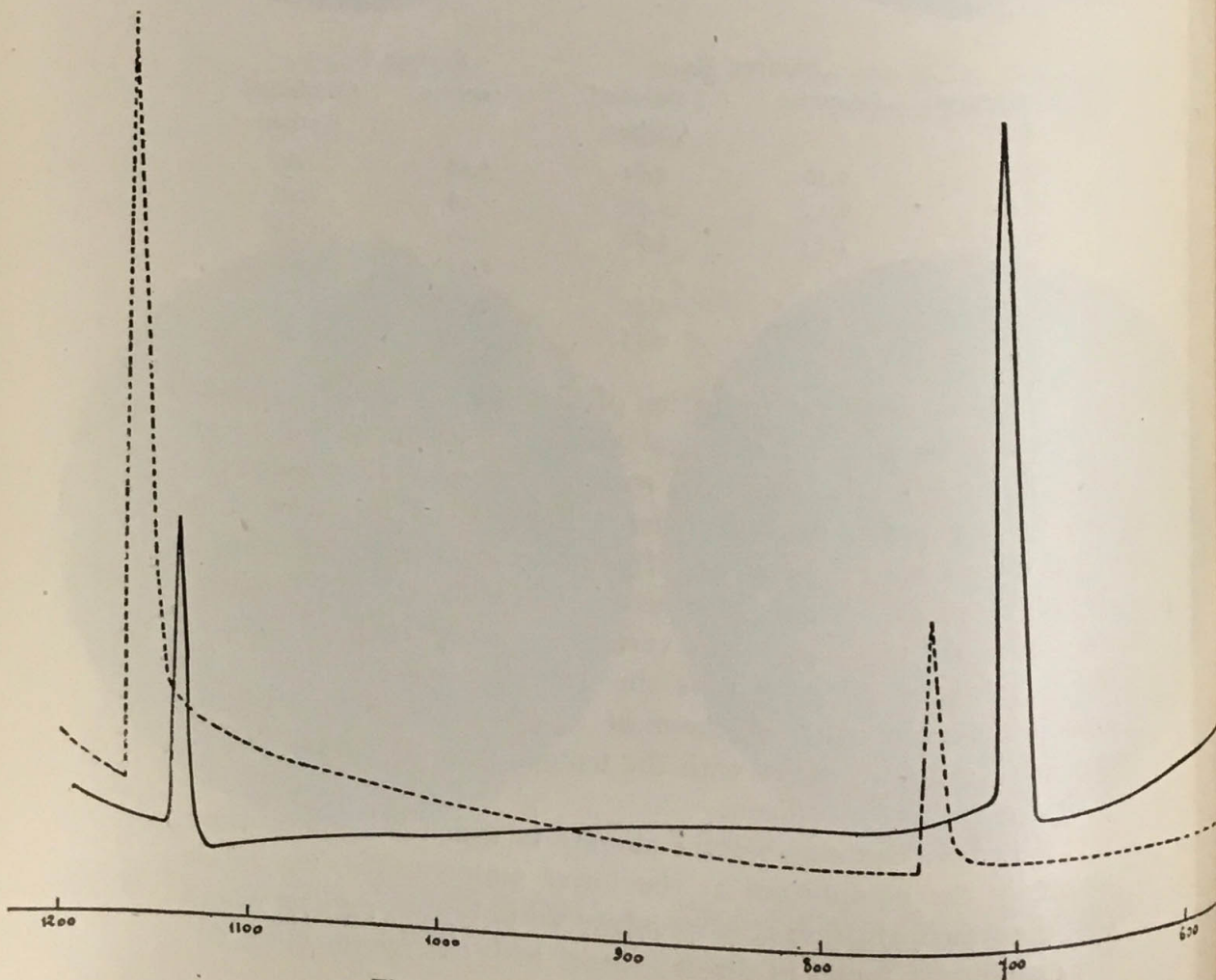


Fig. 7. ----- Heating. ——— Cooling.

It is natural enough to admit that these are the regions where the transformation is complete: consequently, the stable state in the cold of iron-carbon systems corresponds to the coexistence of the two phases, pure iron and graphite; all the other forms obtained in iron and steel, notably cementite, would be unstable at lower temperature. The case of iron-carbon systems is to be compared to that of phosphorus, for which the stable form in the cold is red phosphorus, but what is obtained and used most often is the unstable form of white phosphorus.

We have also determined the critical points of the irons studied by the pyrometric method. For these different samples we have always observed two very distinct critical points, practically at the same temperatures. The first, which occurs at about 700 degrees, corresponds to the resolution of the pearlite. It is too well known to be explained here. The second is observed at about 1150 degrees: it is extremely distinct, as shown in Fig. 7, which represents the heating and cooling of an iron poor in silicon. It is produced in the same conditions with silicious and non-silicious irons.

The following table shows the temperatures at which this second critical point has been observed for the different samples:

Sample	Heating	Cooling
No. 1	1140	1120
No. 2	1165	1145
No. 3	1137	1130
No. 4	1165	1137
No. 5	1165	1145

It has not been possible for us to determine if this critical point corresponds to the resolution of the martensite-graphite eutectic mixture, or to that of the martensite-cementite eutectic mixture. Perhaps it may combine the two transformations, which would occur at much closer temperatures than indicated by Roozeboom's diagram. A very interesting peculiarity is that this critical point of 1150 degrees is, in all the samples, much more emphasized in the heating than in the cooling. It is the inverse of what is ordinarily observed with critical points.

The observations contained in this note already indicate that a certain number of points in Roozeboom's diagram require modification; but it seems necessary to make additional experiments before suggesting a new chart.



## IRON AND STEEL FROM THE POINT OF VIEW OF THE "PHASE-DOCTRINE" \*

By Baron HANNS JÜPTNER VON JONSTORFF

THE several, and to some extent self-contradictory, theories expounded by Professor Bakhuis-Roozeboom† in his epoch-making publication of last autumn have induced me to reëxamine the original treatises of which he made use in support of his views. My object in so doing is to aid, if possible, in establishing a definite basis for future experimental research, and it is my intention now to deal principally with the state of equilibrium between martensite and graphite.

The following table shows the results of Mannesmann's cementation experiments, after making due allowance for the correction of temperatures. It should be mentioned that in calculating the latter, precisely the same method has been followed as that employed by H. Le Chatelier‡ in dealing with the temperatures of the fusion of substances as given by Mannesmann:

Percentage of Carbon	Temperature of Saturation	Percentage of Carbon	Temperature of Saturation
	° C.		° C.
0.35	400	3.5	1060
0.5	760	4.0	1070
1.0	890	4.5	1080
1.5	960	5.0	1085
2.0	990	5.5	1090
2.5	1020	6.0	1095
3.0	1040	6.5	1100

On the other hand, Mannesmann states that iron, when at the melting-point of copper, say at about 1050° C., can absorb only 1.8 per cent of carbon, a statement strikingly at variance with the data given in the table. Osmond,§ moreover, suggests with good reason that Mannesmann very possibly never reached the saturation point.

\* *Iron and Steel Institute*, 1901, Vol. I.

† *Journal of the Iron and Steel Institute*, 1900, No. II, p. 311.

‡ *Bulletin de la Société d'Encouragement*, 1900, p. 661.

§ *Bulletin de la Société d'Encouragement*, 1900, p. 652.

In some further experiments on the cementation of iron carried out by Royston,\* the following values representing the rate of absorption of carbon were obtained:

At 620° C.	.	.	.	.	.	0.0	per cent carbon
" 720° C.	.	.	.	.	.	0.85	" "
" 1030° C.	.	.	.	.	.	1.50	" "

But unfortunately these results can only be regarded as dubious when they are contrasted with the experiments of Mannesmann, which show that iron could take up 2.75 per cent carbon at a temperature of 1030° C.

Royston next heated a sample of white iron containing 3.85 per cent carbon to various degrees of temperature, allowing it to cool gradually. The iron, when analyzed, was found to contain:

	Combined Carbon Per Cent	Graphitic Carbon Per Cent
At 670° C.	1.10	2.75
" 720° C.	1.20	2.65
" 740° C.	3.05	0.80

Thus it is seen that the percentage of carbon which remained in the combined state here is much in excess of that obtained in his former series of experiments, though this difference is probably due to the incomplete decomposition of the carbide at the different temperatures.

Continuing his experiments, Royston next rapidly heated white iron containing 3.85 per cent carbon to 1030° C., and allowed it to cool gradually; a second sample of the same iron was similarly heated to 1030° C. and suddenly quenched in water. He obtained the following results:

	Combined Carbon Per cent	Graphitic Carbon Per cent
After gradual cooling	1.50	2.30
After quenching	1.50	2.35

In these experiments it is remarkable that the proportion of combined carbon in each case is exactly equal. The second experiment might have assisted in throwing some light on the doubtful conditions of equilibrium if it could have been shown that a complete dissociation of the carbide had taken place.

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\* *Journal of the Iron and Steel Institute*, 1897, No. I, p. 166.



Turning now to the experiments of Saniter,\* more important results are to be noted. He heated a piece of iron wire of very pure quality, 0.04 inch in diameter, in a porcelain tube together with charcoal. This was subjected for varying periods to a temperature of 900° C., and after being allowed to cool in the tube, a portion was removed for analysis. It was found to contain:

	Original Wire	After 7 hours heating	After 14 hours heating	After 21 hours heating
Total Carbon . . .	Trace	Per Cent 1.64	Per Cent 2.79	Per Cent 2.95
Graphitic Carbon .	—	—	—	0.53
Combined Carbon .	—	—	—	2.42
		Pearlite with bands of cementite, extending in all directions to the surface.		Pearlite, cementite, and graphite (the latter could not be detected under the microscope). The cementite was segregated towards the centre, none of the bands extending to the exterior; the surface of the wire was coated with graphite, which was carefully removed before analysis.

The rate at which the carbon was absorbed was as follows:

In the first 7 hours . . . . .	1.64 per cent
“ second 7 hours . . . . .	1.15 “
“ third 7 hours . . . . .	0.16 “
Total . . . . .	2.95

from which Saniter deduced that at 2.95 per cent the saturation point was reached.

According to Professor Arnold† the diffusion of carbon begins only at 750° C., and increases suddenly when 900° C. is reached. He states that there diffuses:

At 750° C. . .	Fe <sub>2</sub> C (up to 0.9 per cent Carbon).
At 900° C. . .	Fe <sub>3</sub> C (up to 2 per cent Carbon at the least).

\* *Journal of the Iron and Steel Institute*, 1897, No. II, p. 122.

† *Journal of the Iron and Steel Institute*, 1897, No. I, p. 101.

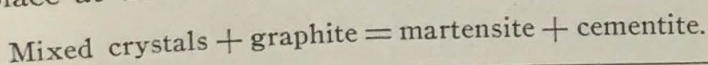
Following the same line of investigation, Margueritte\* heated finely divided iron, which he had obtained from oxalate, for three hours consecutively in a current of carbonic oxide gas, and then found that the following quantities of carbon had been absorbed:

	Carbon Percentage
At dull red heat, 635° C.-694° C. (Taylor) . . . . .	6.60
At bright red heat, 843° C. (Taylor) . . . . .	6.55
At dark orange, 950° C. (fusing point of silver) . . . . .	1.21

In this connection Osmond points out with reason that it cannot possibly be due to mere accident that the formation of carbide was effected in the first two instances.

It is evident, then, that, with regard to the process of cementation, results were obtained by the several investigators which, while differing in the extreme as to the carbon contents, are in striking accord in the result that the cement steel after the first heating was always found to contain carbon in the combined form alone, and it was only after the second, or at any rate after a prolonged heating, that the formation of graphite occurred. It is abundantly clear that there are thus two entirely distinct phenomena.

Particularly instructive are Saniter's experiments, which demonstrate that within fourteen hours of the commencement of the cementation process the steel contained only martensite and cementite, and that the cementite bands penetrated the entire mass as far as the surface. At the end of twenty-one hours, however, the cementite had segregated near the centre, and graphite had been formed. These successive phenomena point to the conclusion that within a temperature of about 1000° C. graphite is not the more stable form of carbon compound. This view is also strongly borne out by Osmond's† observations, which, taken in conjunction with my own views‡ on the critical points in the neighborhood of 1000° C., would seem to furnish ample proof of the existence of the reaction assumed by Roozeboom to take place at this temperature:



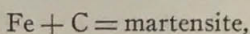
\* *Comptes Rendus*, Vol. LIX, p. 726.

† *Bulletin de la Société d'Encouragement*, Vol. V, 1900, pp. 657, 658.

‡ *Journal of the Iron and Steel Institute*, 1900, No. I, p. 219.

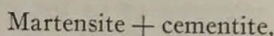


It must, therefore, be assumed that the absorption of carbon by the iron at the temperature in question is accompanied by the formation of martensite, — and that in the first place the state of equilibrium,

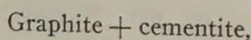


though perhaps only apparent, is established.

In such cases where the martensite does not come into immediate contact with the carbon, that is to say, in the interior of the mass of iron or at such places on the surface where no charcoal actually touches, the separation of cementite may occur, and indeed must, when the point of saturation corresponding to the given temperature is reached, and thus there is reached a second state of equilibrium,



which not only explains the high carbon percentage which some investigators obtained, but agrees fully with the micrographic observations recorded by Saniter. Whether the carburisation under these circumstances can be continued till the state of equilibrium,



is reached, is open to question, but the theoretical possibility of reaching it cannot be denied. Evidence of this is also afforded by the experiments of Margueritte.\* If this were possible, the corresponding equilibrium curve would be represented by a line drawn vertical to the carbon percentage line at 6.67 per cent carbon.

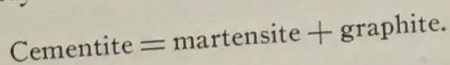
In explanation of the occurrence of graphite after long heating to redness or prolonged cementation, it will be necessary to consider Saniter's experiments on the behavior of the carbide of iron at higher temperatures. Briefly the results were as follows:

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\* It is, however, quite possible that the high degree of carburisation obtained by Margueritte was mainly the result of the use of carbon monoxide, instead of carbon in a solid form.

	First sample		Second sample	
	Combined Carbon	Graphitic Carbon	Combined Carbon	Graphitic Carbon
	Per Cent	Per Cent	Per Cent	Per Cent
Original Carbide . . . . .	6.73	0.19	6.46	0.22
After heating in nitrogen to 800° C. and gradually cooling } . . . . .	5.72	0.40	—	—
After heating in nitrogen to 1000° C. and gradually cooling } . . . . .	3.63	2.50	3.79	2.41
After heating to 1000° C. and then quenching } . . . . .	5.57	0.56	—	—
Fused with magnesia and allowed to cool gradually } . . . . .	—	—	1.22	3.05

It will be noticed that with the increase of temperature the carbide dissociates; this is shown too in Margueritte's experiments, where the diminution in the amount of carbon absorbed is apparent as the temperature increased; it is also evident that the dissociation of the cementite must commence when martensite is present, probably according to the equation —



This theory still leaves much to be accounted for; for instance, the formation of graphite in conjunction with ferrite in the Brustlein steel, described by Osmond,\* and further investigation of which is necessary before any definite conclusion can be drawn.

Above the temperature under consideration, of about 1000° C., at which the transformation of graphite + martensite into cementite occurs, there can exist no doubt that graphite has a greater stability than cementite. The formation of graphite in slowly cooled iron seems to confirm this, as well as the dissociation of the cementite. But if this is so, then the line *a E*, which, according to Roozeboom's hypothesis, indicates the separating out of the graphite at the corresponding temperature, must cut the cementite curve at the point *E*.

\* In regard to this, particular attention should be directed to the influence of other elements.



Of great interest are Le Chatelier's observations on the expansion of steel and the formation of troostite. The sudden contraction attending the transformation of pearlite into troostite would account for the diminution of volume which so frequently occurs during the formation of solutions. In like manner it would seem reasonable to assume that the equally sudden expansion which accompanies the transformation of troostite into martensite may be accounted for by regarding troostite as a solution of carbide of iron, which is decomposed or becomes depolymerized on its transition into the martensite form; the expansion being due to the increase in the number of existing molecules.

## THE EFFECT OF REHEATING UPON THE COARSE STRUCTURE OF OVER-HEATED STEEL \*

By K. FREDRIK GÖRANSSON

### INTRODUCTION

THE structure of steel, when rendered coarse by over-heating, is made fine by reheating to a certain temperature, the determination of which has received much attention from eminent metallurgical authorities. Among these I may particularly mention Professor D. Tschernoff, who, about thirty years ago, studied this question in connection with his investigations on the structure of steel in general.†

Some years later, Mr. J. A. Brinell took up the inquiry, and

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\* *Transactions American Institute of Mining Engineers*, 1902.

SECRETARY'S NOTE. — This paper is substantially a thesis presented by the author in May, 1901, to the Faculty of Science of Columbia University, in New York. By consent of the Council, it is published also in the Swedish language by *Jernkontoret* of Sweden. Members of the Institute will be interested to know that the author is the grandson of G. F. Göransson, whose experiments and improvements contributed essentially to the first practical success of the Bessemer process. (See the paper of Professor Akerman, *Transactions*, Vol. XXII, p. 265.) R. W. R.

† "On the Manufacture of Steel and the Mode of Working It." (May, 1868.) Translated in *Proceedings Institute of Mechanical Engineers*, January, 1880.

arrived at many important conclusions, stated in his well-known rules on the treatment of steel.\*

These two gentlemen, however, based their reasoning chiefly upon the aspect of fractures — a method which, though it affords valuable information, does not readily reveal the different steps by which a change in structure takes place.

For further light on the subject we owe much to more recent microscopic investigations, which have made us better acquainted with the internal structure of metals. Such an investigation, closely connected with the present subject, and made by Mr. John E. Stead a few years ago, was first published in 1898.†

Regarding the cause of the disappearance of the coarse structure, as the result of annealing, Mr. Stead's observations led him to conclusions somewhat modifying, or, at least, rendering more complete, those of earlier investigators.

Mr. Brinell found that the refinement of structure takes place at the same temperature as the transformation of the cement-carbon into hardening-carbon, and concluded that the very violence of the transformation destroys all previous crystallization.

Mr. Stead, however, in examining the micro-structure of a piece of over-heated iron, containing 0.11 per cent of carbon, which had been reheated to  $830^{\circ}$  C. and then slowly cooled, found evidences of another factor, namely, the *diffusion of the carbon* into the grains of ferrite. He says:\*

"... The pearlite was redistributed between the grains, and it could be easily seen to what point the carbon had diffused... Where the carbon has diffused into the ferrite, its coarse structure is broken up. We conclude from these results that the breaking up of the coarse structure in carbon-steel is not due only, as Brinell maintains, to the coincident change of cement- into hardening-carbon, but also to the carbon diffusing after such a change has been effected."

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\* "Textual Changes Effected in Steel by Heating and Cooling." *Jernkontorets Annaler*, 1885, Vol. XL, pp. 9-38. Abstracted in *Journal of the Iron and Steel Institute*, 1886, No. 1, pp. 365-367. German translation in *Stahl und Eisen*, Vol. V, p. 611.

† "The Crystalline Structure of Iron and Steel," *Journal of the Iron and Steel Institute*, 1898, pp. 145-189; also, *The Metallographist*, October, 1898.

‡ *Loc. cit.*, p. 166.



The aim of my investigation has been to collect additional facts which might be useful in the further study of this problem.

The steel I studied was a Swedish Bessemer steel, rolled to bars of  $\frac{8}{16}$  by  $\frac{9}{16}$  inch. Its composition was:

	Per Cent
Carbon . . . . .	1.200
Phosphorus . . . . .	0.028
Silicon . . . . .	0.030
Sulphur . . . . .	0.002
Manganese . . . . .	0.230

The plan pursued in the experiment was: 1. To find the critical range of the steel; 2. To overheat it, in order to get a coarse structure; 3. To reheat it to various temperatures, at and about the critical range; 4. To study the effect of these heatings upon the micro-structure.

## I. HEATING- AND COOLING-CURVES

In order to find the *critical range* of the steel, several heating- and cooling curves were plotted. Some of these curves are shown in Figs. 1 and 2. Only one retardation-point could be detected within the range of the experiment ( $575^{\circ}$  to  $1060^{\circ}$  C.). This retardation took place at  $640^{\circ}$  to  $755^{\circ}$  C. in heating, and at  $690^{\circ}$  to  $700^{\circ}$  C. in cooling, and seems to indicate the point " $A_{2-1}$ "; that is, the temperature at which the eutectic pearlite changes into martensite, and *vice versa*.\*

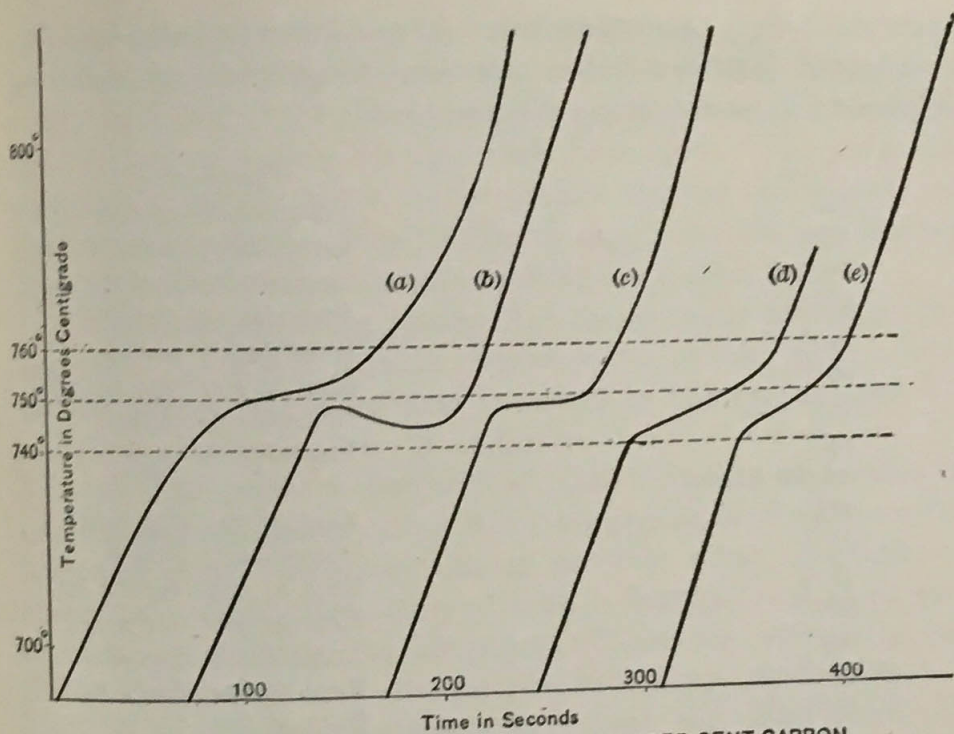
## II. OVERHEATING

For overheating, the bar was cut up into pieces of 3 inches, heated to above  $1270^{\circ}$  C., and then cooled slowly with the fur-

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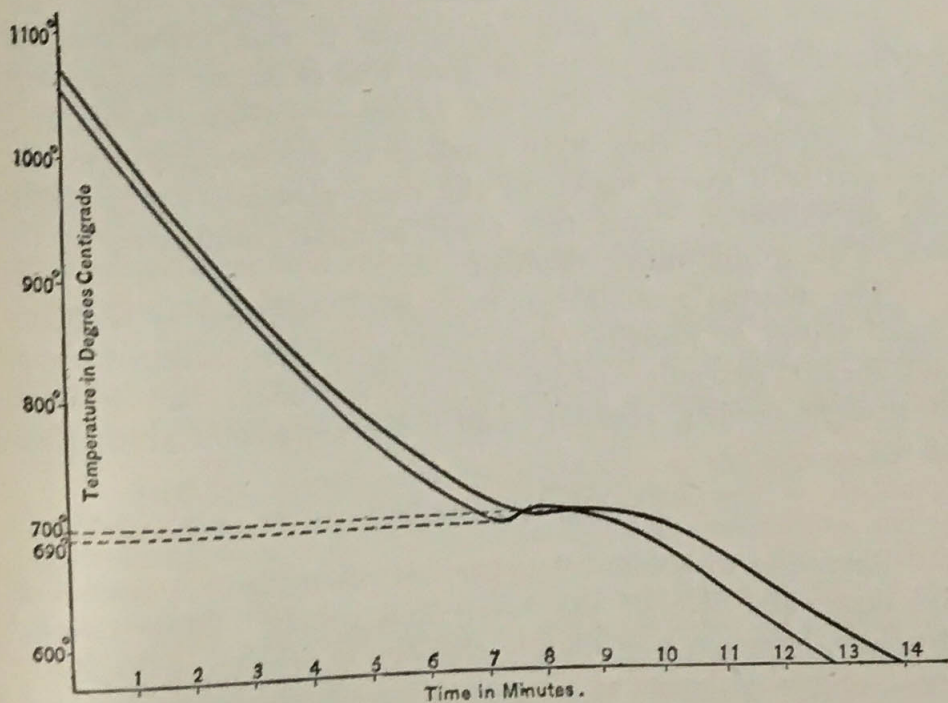
\* In connection with these curves, I would say that in the case of one of the readings for the heating-curve (curve *b*, Fig. 1), the pyrometer indicated a *fall* of temperature at the retardation-point. The movement of the pyrometer was steady, and I do not think this apparent fall was caused by any defect of the instrument. In this special case  $Ac_1$  seems to have been somewhat delayed; and perhaps this fact caused the transformation to be more violent than usual.

I do not think it would be proper to make general inferences from the appearance of these few curves; but I desire to point out that in those cases (*b* and *c*, Fig. 1) where the retardation began at a comparatively high temperature, it was completed at a comparatively low temperature.



HEATING-CURVES OF STEEL CONTAINING 0.20 PER CENT CARBON

Fig. 1.



Cooling-Curves of Steel Containing 0.20 Per Cent Carbon.

Fig. 2.



nace, reaching a black heat after three-quarters of an hour. The overheated steel was then cut into lengths of 1.5 inch, and reheated to the following temperatures:

T <sub>max</sub>	Treatment
920° C., . . . . .	Immediately cooled.
887° " . . . . .	Immediately cooled.
850° " . . . . .	Immediately cooled.
816° " . . . . .	Immediately cooled.
790° " . . . . .	Immediately cooled.
765° " . . . . .	Kept at 760° for 1.5 hr.
760° " . . . . .	Immediately cooled.
748° " . . . . .	Immediately cooled.
(Acl.)	
725° " . . . . .	Kept at 725° for 2 hrs.
725° " . . . . .	Immediately cooled.
712° " . . . . .	Kept at 695° for 1.5 hr.
680° " . . . . .	Kept at 660° for 2 hrs.
675° " . . . . .	Immediately cooled.
675° " . . . . .	Kept at 670° for 2 hrs.
625° " . . . . .	Kept at 625° for 2 hrs.
575° " . . . . .	Kept at 575° for 2 hrs.

The coolings were made naturally in the air.

In examining the micro-structures it was found that the overheating had changed the original fine structure into a coarse net-work of cementite. The size of these polygons varied very much. In general they were smallest in the center of the bar; but even in a given region of the same specimen the variation was considerable. This fact made accurate measuring of the size of the grains rather difficult.

The internal structure of the grains consisted of well-laminated flakes of pearlite, with, here and there, isolated lumps and lines of cementite, which, in some specimens, had the form of straight needles, crossing each other apparently at an angle of 60°.

### III. REHEATING

Reheating to points below  $A_{c1}$  seemed to have no effect on the shape and size of the overheated grains. Their internal structure, however, was slightly changed in specimens "soaked" for a couple of hours at a temperature closely below  $A_{r1}$  (but not at lower temperatures). This change consisted in the formation of rings of cementite, isolating small lumps of pearlite in

the interior of the grains. It would seem that these rings were caused by a flow of the cementite, which, during the soaking, might have had more time to arrange itself than it had during the continuous cooling of the overheated piece. The rings had collected in certain parts of the grains, leaving other parts apparently unchanged. The laminated aspect of the pearlite was apparently the same after this soaking as before.\*

When the reheating passes  $Ac^1$  the changes are more pronounced. A twofold transformation seems to take place: (a) a sudden change in the internal structure of the grains; and (b) a gradual change in their boundaries.

(a) The previous lamination of the pearlite is broken up and the new pearlite is rather badly laminated, if it can be called laminated at all. Etching with 20 per cent nitric acid makes it appear as a dark mass, covered with a multitude of small dots of cementite, looking like pin-holes. These dots are pretty uniformly distributed over the surface, but many of them show a tendency to arrange themselves into rings and short rows. In some places, even, the dots unite, making continuous lines instead of dotted ones; but this is not generally the case. Only when the reheating temperature passes  $800^\circ$  C. do they show a stronger tendency to melt together. In that case the union of some of the dots may effect a more definite lamination of the pearlite, while others form a net-work of the cementite, enclosing a new set of grains inside the old ones. These new grains are rather small and irregular; but as the temperature rises, they grow larger and assume a polygonal form, accompanied by a more regular lamination of the pearlite.

(b) As to changes produced in the boundaries of the old grains, it may be said that, if reheating barely passes the retardation point, it has no appreciable effect on the old net-work of

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\* About nine months after the completion of the experiment here described, a piece of over-heated, but not reheated, steel, which had not exhibited such rings upon the first examination, was reexamined, and was then found to contain them, although to a smaller extent than was shown in the reheated specimens. The only new manipulation which this piece had undergone was repolishing and reetching—the latter being performed with dilute nitric acid, in the same manner as before. This observation, if confirmed by similar ones, would indicate that the formation of the cementite rings takes place in cold steel, if sufficient time be allowed for it.



cementite; but as the temperature rises still higher, the net-work becomes disconnected and loses itself in the finer net-work of the new grains above described. This change is very gradual and is not completed until a temperature of more than  $100^{\circ}$  C. above the retardation point has been reached. That the transformation is a question of temperature rather than time, is shown by the fact that, in specimens soaked at  $760^{\circ}$  C. for 1.5 hours, the old net-work was still well preserved.

#### IV. THEORETICAL INFERENCES

The above observations seem to indicate that the destruction of the coarse net-work of cementite is caused by its carbon being dissolved in the martensite, and that the net-work surrounding the new grains is formed by the expulsion of cementite from the martensite as it is being cooled.

It is important to distinguish between the formation of the *grains* and the formation of the *net-work* of cementite between them, for it is probable (and is generally held) that the grains exist in the martensite even at the highest temperatures, and that they are caused by a tendency of the particles of martensite to arrange themselves in certain directions. It also seems that the higher the temperature, the more effect has this tendency, and the larger are the resulting grains.

During the cooling, cementite is expelled from the martensite and seems to collect between the grains, broaden the boundaries, and enable us to detect them by the microscope.

Now, the cementite expelled in the overheated steel forms a much coarser net-work than that expelled in the reheated steel. This fact may indicate that the polarization of the martensite into coarse grains has been destroyed between the two processes of expulsion.\*

When should this destruction of the polarization have taken

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\* A fact which possibly might indicate a destruction of this polarization is the dotted or imperfectly laminated aspect of pearlite formed in cooling from temperatures slightly above the retardation-point. We do not yet know the cause of the lamination of pearlite; but if it has something to do with a polarization of the martensite, it would seem natural that an imperfect polarization, such as might be produced at the lower temperatures, would go hand-in-hand with an imperfect lamination of the pearlite.

place? It seems to me that we have two possibilities: Either the destruction of the polarization might have taken place when the reheating reached  $Ac_1$  — in which case we approach the view held by Mr. Brinell; or the destruction of the polarization might have already taken place when the overheated steel, during its cooling, passed the recalescence point. According to this hypothesis, the coarse net-work of cementite was then already formed, and served to preserve the coarse structure of the steel, even though the polarization of the particles no longer existed.\*

## V. PRACTICAL CONCLUSIONS

To the practical question, What reheating temperature is needed in order to break up the coarse structure? the reply, on the above theory, would be that we have to heat the steel to the point where *all* the cementite has been dissolved by the martensite. And this seems to be, in fact, the case; for, in the experiments here described, where the steel had been reheated to  $850^\circ \text{C.}$ , there were many of the old large grains remaining; but where the reheating had reached  $887^\circ \text{C.}$ , the large grains had completely disappeared.

According to the curves of Sir W. Roberts-Austen and of Professor Roozeboom,† the cementite of a 1.20 per cent carbon-steel begins to fall out from the martensite at about  $870^\circ$  to  $880^\circ \text{C.}$ , that is, at a temperature intermediate between the reheating temperatures of  $850^\circ \text{C.}$  and  $877^\circ \text{C.}$ , determined in these experiments.

Whether this theory is valid for hypo-eutectic steels also, future experiments must decide. It may, however, be of interest to point out that Mr. Stead, in the experiments on the crystalline structure of iron and steel, reported in his paper, already cited, found a refinement of the grains at  $900^\circ \text{C.}$  for steel of 0.01 per cent, and at  $870^\circ \text{C.}$  for steel of 0.11 per cent carbon, and that these two points correspond to the temperatures at which, for such steels respectively, the ferrite begins to fall out from the martensite.

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\* This supposition is favored by the rearrangement of the cementite, already mentioned, in specimens reheated to points closely *below*  $Ac_1$  and kept there for some time.

† *The Metallographist*, April, 1901, p. 154.



## POSTSCRIPT

BY HENRY M. HOWE, NEW YORK CITY

The work described in this paper was undertaken as a step toward studying the coarsening of the grain of steel which takes place as the temperature is progressively raised above the critical range, and the refining of the grain by reheating to that range. A recapitulation of the points shown by the investigation will facilitate an explanation of their significance.

When steel, the grain of which has been made extremely coarse by greatly overheating it, is cooled and subsequently reheated (for instance, as a step in annealing), the following changes occur:

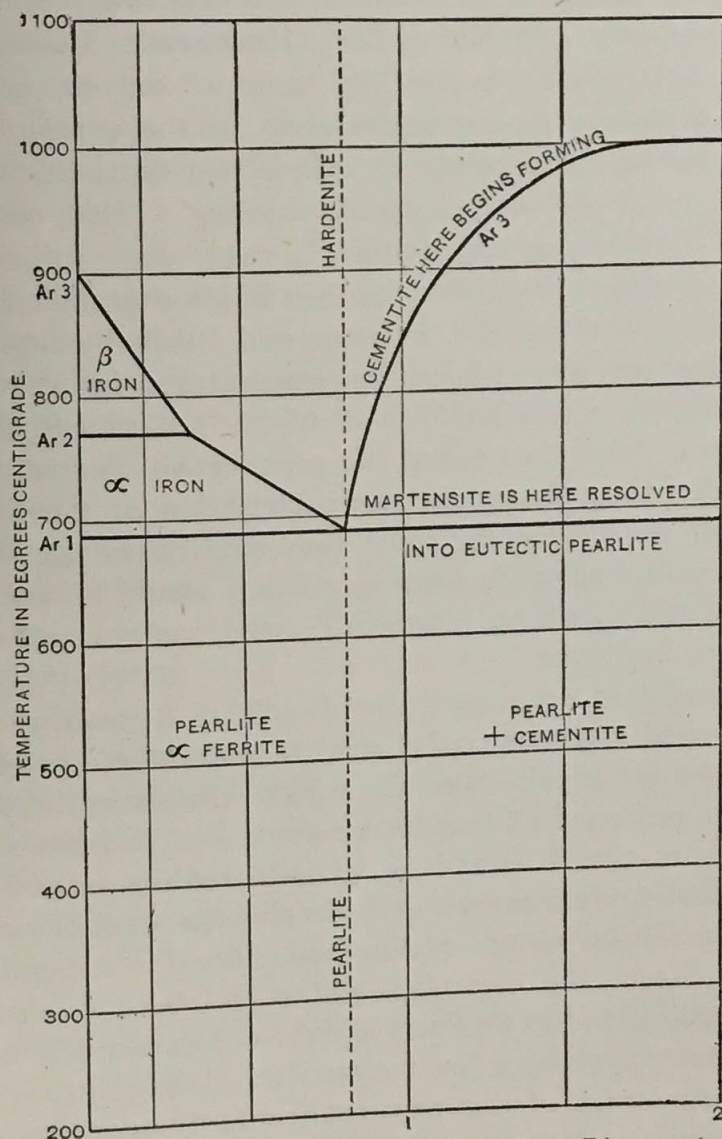
1. *Segregation below  $Ac_1$ .* — At a temperature slightly below  $Ac_1$ , cementite seems to segregate into rings, apparently because of surface tension. Mr. Göransson has furnished, so far, only slight hints as to this action.

2. *Behavior of the Net-Work of Cementite.* — If the temperature is carried above  $Ac_1$ , the cementite net-work is progressively re-absorbed by the grains of martensite which it surrounds. A portion of Roberts-Austen's diagram, reproduced in Fig. 3, shows that, in the cooling of such hyper-eutectic steel from about  $950^\circ$  ( $Ar_3$ ) to  $690^\circ$  ( $Ar_{2-1}$ ), in which range the mass of the material is in the condition of martensite, the solvent power of this martensite for cementite progressively diminishes, so that cementite progressively separates out within the martensite, forming itself into a net-work, the coarseness of which is proportionate, roughly speaking, to the temperature to which the steel had been previously heated.

Now, the re-absorption of the net-work of cementite, which Mr. Göransson has observed, is simply the converse of this. As the temperature rises from  $Ar_{2-1}$  toward  $Ar_3$  the solvent power of the martensite progressively increases; that is, the martensite is able to redissolve more and more of the previously formed cementite net-work. This progressive increase in the solvent power of the martensite for cementite, and the simultaneous increase in the case of diffusion with rise of temperature, suffice to explain readily the progressive re-absorption and final disappearance of the net-work of cementite. Mr. Göransson's ob-

servations in this respect confirm earlier (unpublished) observations of my own.

When hyper-eutectic steel is heated above the critical range, and thus passes into the condition of martensite, and is then



Cooling-Curves of Hyper-Eutectic Steel, from Diagram by Roberts-Austen.

Fig. 3.

subsequently cooled and thus changed back into pearlite and cementite, the net-work of the cementite is coarser, the higher the temperature to which the steel has been heated. It is an important question whether this progressive coarsening of the cementite net-work is due to progressive change in the polarization



of the martensite or in that of the cementite itself. It is true that in martensite that is free from this net-work of cementite it has not yet been found possible to distinguish this progressive coarsening. In my opinion, however, this is only because we have not yet learned how to detect the coarseness of the grain in the martensite. So far as Mr. Göransson's observations go there is nothing to show that the cause of coarsening is not a progressive change in the polarization of the cementite, corresponding to the temperature at which the cementite begins to separate out of the martensite in cooling. This temperature would be  $T. \max.$ , provided that  $T. \max.$  does not rise above  $Ar_3$ , as may indeed have been the case in his experiments. However, in the steel which Mr. Sauveur and I examined,\* we found that this coarsening continued at temperatures far above  $Ac$ , — at temperatures, consequently, at which no cementite existed. In my opinion this shows that the progressive coarsening of the grain is due to a change in the polarization of the martensite and not of the cementite, since the temperature at which the cementite would begin to form in cooling would be one and the same, viz.,  $Ar$ , no matter whether  $T. \max.$  has been only slightly or very far above  $Ac$ .

3. *Formation of a New Net-Work of Cementite.* — When the overheated and then cooled steel is reheated to between  $Ar_{2-1}$  and  $Ar_3$ , for instance to  $800^\circ C.$ , part of the cementite, which in the former cooling had become insoluble and had therefore separated out between  $800^\circ$  and  $Ar_{2-1}$ , again redissolves and diffuses into the surrounding martensite. When the steel is now again cooled, the solvent power of the martensite for cementite again decreases; hence the cementite which had been re-absorbed is now reëxpelled, and in this new expulsion forms a new net-work. It is in accordance with the views which I have just expressed that the coarseness of this new net-work of cementite increases with  $T. \max.$

4. *Blurring and Subsequent Reclearing of Pearlite.* — Mr. Göransson observes that if the steel be heated only slightly above  $Ar_{2-1}$ , the pearlite found in it after recooling is blurred. If, however, the heating be carried somewhat higher, the pearlite is again cleared. An obvious explanation suggests itself. The

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\* See *Eng. and Min. Jour.*, Vol. LX, Dec. 7, 1895, p. 537.



original pearlite was formed through slow cooling from  $Ar_{2-1}$  downward, which splits up the martensite into alternate layers of ferrite and cementite. On reheating past  $Ac_{1-2}$ , these tend to recombine to form martensite again; but at the relatively low temperature at which this occurs, and in the relatively rigid state of the metal, the recombination and diffusion of the sheetlets of ferrite and cementite into each other are relatively slow. If we arrest the heating when the temperature has passed only very little above  $Ac_1$ , this diffusion is incomplete, so that traces of the former pearlite structure remain in red-hot metal. When this now cools below  $Ar_{2-1}$ , and the martensite formed by this imperfect diffusion again resolves itself into pearlite, we can hardly expect that the new pearlite will be well laminated; for such good lamination should occur only when the original mass in which the pearlite forms is approximately homogeneous. Under the conditions which we have been assuming, the structure of the old pearlite may be expected to cross that of the new. Consider two adjoining sheetlets of the original ferrite and cementite; suppose that, with the heating past  $Ac_1$ , they had diffused so far that but one-quarter of them had reconstituted martensite of the pearlite composition (viz., 0.80 per cent carbon), but that on either side of this band there were regions of hyper-eutectic and hypo-eutectic martensite respectively. In their recombination, then, these two sheetlets will have formed at this moment a flake of martensite of very heterogeneous composition; and this heterogeneousness is quite sufficient to explain why, when the martensite again splits up into pearlite, that pearlite should be irregular, as reported by Mr. Göransson, instead of being well-laminated.

As the distance above  $Ac_{1-2}$  to which the reheating is carried is progressively increased, so should the diffusion of the ferrite and cementite, and the homogeneousness of the resultant martensite, increase; and so should the regularity of the pearlite, which, in turn, results from the re-decomposition of that martensite on again cooling, increase. Thus may the blurring and reclearing of the pearlite be explained.

The explanation of the clouding and clearing of the pearlite can be tested in a simple way, which will probably be carried out in my laboratory. It is as follows: Heat a series of specimens of steel to temperatures varying progressively from barely above



$Ac_1$  to a temperature high enough to bring about the clearing of the pearlite; but, instead of cooling them slowly so as to form new pearlite, quench them in water. If my explanation is correct, then those quenched from temperatures only above  $Ac_{2-1}$  should exhibit traces of the old pearlite structure, whereas those quenched from higher temperatures should show no such traces. Osmond has given the first indications in this direction in his masterly *Méthode Générale*,\* in which he figures the diffusion of the ferrite and cementite into each other in passing through the stage of sorbite.

5. Mr. Göransson's observation, that the polygons are smaller in the center than toward the outside of the specimens, agrees with older observations on fracture, and tends to bring fracture and micro-structure into harmony.†

## PRACTICAL PROBLEMS IN THE METALLOGRAPHY OF STEEL ‡

By J. O. ARNOLD

SINCE Sorby in 1864 established the all-important fact that steel must be regarded as a crystallized igneous rock, his work has been greatly expanded by the international labors of many able microscopists. Much of the work done, however, has been of academical rather than of practical interest, and busy steel works' metallurgists, appalled by the rapid growth of constituents ending in "ite," of "eutectics" and of solid solutions of carbon or carbides in unisolated allotropic modifications of iron, are already beginning to ask themselves the question, Is micrographic analysis going to be of any real use to us, and if so, in what direction? The present article is an attempt to very briefly answer the above questions.

The theory prevalent a quarter of a century ago that steels of identical chemical composition would necessarily have the same mechanical properties has long since been discarded. But per-

\* *Bull. Soc. d'Encouragement*, May, 1895, pp. 35-61 of extract.

† Compare Metcalf, *Trans. Am. Soc. Civ. Eng.*, XV, p. 388, and the writer's *Metallurgy of Steel*, p. 176.

‡ *Nature*, April 25, 1901.

haps steel metallurgists have not yet fully realized the disconcerting fact that steel of excellent chemical composition, giving highly satisfactory mechanical tests, may nevertheless utterly fail in use, possibly with disastrous results. In other words, a ductile steel which bends double cold without any sign of flaw or failure may, under the influence of vibration, snap like a piece of glass, though only subjected to mechanical stresses well below its elastic limit.

In connection with the materials of construction used for high speed engines, both land and marine, it is at the present time a problem of paramount importance for the scientific steel metallurgist to determine the cause of the sudden infidelity of steel (or wrought iron) under vibration.

Data in the writer's possession prove beyond all doubt that steel giving splendid chemical and mechanical tests may rupture under vibration possibly in a few hours or perhaps only after the lapse of twenty years.

There is little doubt that in many cases the microscope is capable of giving warning of the dangerous character of a steel, chemically, and apparently mechanically, safe. To describe intelligibly the structures of safe and dangerous steels it is necessary to consider:

- (a) The micrographic constituents of structural steel.
- (b) The molecular migrations of these constituents when at a red heat the metallic mass is in a semi-plastic state.

To put the case concretely, the chemical constituents of a typical rail may (in addition to iron) be approximately taken as carbon .40, silicon .05, manganese .90, sulphur .06, phosphorus .06 per cent, together with small percentages of arsenic and copper. The micrographic constituents of such a steel are:

1. The pale, simple constituent ferrite (in this case somewhat impure iron).
2. The dark etching compound constituent pearlite, consisting of mixed granules of iron and of a double carbide of iron and manganese.
3. The dove-grey simple constituent sulphide of manganese,  $MnS$ .

It is important to remember that in manganiferous steels the foregoing constituents are only completely differentiated visually on slow cooling from a full red heat, a fact which at once introduces the vital question of the migration of constituents.



Speaking broadly, it may be said that sulphide of manganese is not, under working conditions, capable of migration to any appreciable extent. Thus it remains to consider only the migrations of the ferrite and pearlite.

The movements of these constituents on heating may be termed "diffusion," and their movements on cooling "segregation."

On heating the typical steel specified to about  $700^{\circ}$  C. the compound constituent pearlite is converted, with absorption of heat, into the simple constituent, martensite, at Osmond's point  $Ac_1$ . Then, passing through Osmond's points  $Ac_{2-3}$ , the constituents ferrite and martensite diffuse one into the other till, at about  $800^{\circ}$  C., molecular equilibrium is eventually established.

If, however, the steel be cooled very slowly, the molecules of martensite and of ferrite will perfectly segregate in the respective proportions of about 45 and 55 per cent. Then at  $Ar_1$ , about  $640^{\circ}$  C., the martensite will decompose into the compound constituent pearlite, which, owing to the presence of manganese, will be granular and not laminated. On the other hand, if the steel is somewhat quickly cooled in air, the segregation of the constituents will be imperfect and the apparent proportion of pearlite relatively large, because, owing to the influence of the manganese present, the phenomenon of constitutional segregation is retarded.

As a matter of fact, the apparently large area of dark pearlite is really an extremely intimate mixture of true pearlite and unsegregated ferrite.

The writer is aware that these statements may provoke theoretical opposition, but they nevertheless describe the observed facts, and by these, and not by theories, the practical metallurgist must be guided.

In a brief article it is difficult to do more than give suggestions, but it is necessary to point out that the majority of published micrographs exhibit a single plane of transverse section. Such representation can give only a very partial knowledge of what may be termed the solid geometry of steel.

To determine this it is necessary, in rolled metals, to make three micrographs in three planes of section at right angles to each other, namely, (a) a transverse section, (b) a longitudinal horizontal section, (c) a longitudinal vertical section. From these



the exact form in which any constituent exists in the mass can be determined.

As an example, the case of the constituent sulphide of manganese may be taken. It must be remembered that .09 per cent by weight of sulphur corresponds to no less than .5 per cent by volume of manganese sulphide, a very appreciable amount for a micro-constituent.

In the original ingot during solidification and cooling the sulphide segregates into roughly globular masses. On reheating the ingot for rolling, the sulphide liquefies and the liquid globules are elongated in the direction of the rolling, and necessarily to some extent in a line at right angles to that direction. Hence in a steel plate, the sulphide may present in the three planes of section above specified the appearances exhibited in Fig. 1, which shows that the sulphide is distributed throughout the steel in the form of irregular, oval laminæ.

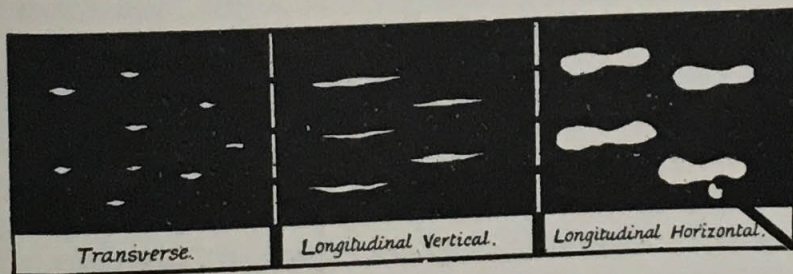


Fig. 1.

It will be obvious that the evil mechanical influence of this constituent will be at its minimum along the length of the plate, somewhat greater across the plate, and at its dangerous maximum through the thickness of the plate.

Perhaps an obstacle to the development of steel works' metallography, even greater than the terror inspired by an unnecessarily complicated nomenclature, is the apparatus, time, care and special re-agents supposed to be necessary to obtain, by polishing and etching, a good micro-section. In the advocacy of this view no one has been more earnest than the writer, and for research purposes it is doubtless sound. But for works' purposes, in connection with most steels, it must be confessed that such necessity has been exaggerated.

The writer has, therefore, pleasure in withdrawing his former view owing to experience having proved that by a very much sim-



plified modification of method, a micro-section may be placed upon the stage for examination in five minutes after it leaves the dead smooth file in the machine shop. This process, which entirely does away with elaborate polishing apparatus or special etching re-agents, is as follows:

Take two pieces of hard wood,  $12'' \times 9'' \times 1''$ , planed dead smooth on one side; then by means of liquid glue evenly attach to the smooth faces two sheets of the London Emery Works Co.'s Atlas cloth, No. 0. Allow the glue to set under strong pressure. Next, by means of a smooth piece of steel, rub off from one of the blocks as much as possible of the detachable emery. This is No. 2 block, the other, necessarily, No. 1 block.

The steel section, say  $\frac{1}{8}$  inch thick and  $\frac{1}{2}$  inch diameter, is rubbed for one minute on No. 1 block, the motion being straight and not circular; then, for the same time and in the same manner rub on No. 2 block. Next place the bright but visibly scratched section in a glass etching dish  $3'' \times 1' \times \frac{1}{2}''$ , and cover the steel with nitric acid, sp. gr. 1.20.

Watch closely, until in a few seconds the evolved gases adhering to the section change from pale to deep brown and effervescence ensues. Then, under the tap, quickly wash away the acid and for a minute immerse the piece in a second dish containing rectified methylated spirits. Dry the section by pressing it several times on a soft folded linen handkerchief, when it will be ready for examination. The structure will be clearly exhibited, the innumerable fine scratches visible before etching having virtually vanished.

The micrographs illustrating this article were prepared in a very few minutes by the above process, and have been accurately reproduced by Mr. F. Ibbotson. The writer hopes that this simple and rapid method may help to stimulate in steel works' practice a more extensive study of metallography.

## MICRO-CRYSTALLINE STRUCTURE OF PLATINUM \*

By THOMAS ANDREWS

THE crystalline structure of platinum does not appear to have been studied, although it forms an interesting subject for investigation.

A small ingot of pure platinum was obtained for the experiments. A section was cut therefrom and machined to  $\frac{5}{16}$  inch square and  $\frac{1}{10}$  inch in thickness. The section was then carefully polished and etched in aqua regia of the following strength:

4 parts of hydrochloric acid (sp. gr. 1.2).  
1 part of nitric acid (sp. gr. 1.42).

When the polished micro-section was immersed in the aqua-regia solution in the cold, no solvent action occurred, although the metal was exposed for a considerable time in the re-agent.

The temperature of the etching solution was then gradually raised to boiling point, at which it was maintained for 15 seconds, but it had apparently no visible effect on the platinum, as ascertained by microscopical examination, after having previously washed the section with water and pure alcohol.

The etching process was repeated and the metal again exposed for a further period of 30 seconds, but after microscopical examination of the surface of the metal it was found that even this exposure had not satisfactorily developed a structure in the metal.

The etching process was again repeated and the micro-section exposed for another 15 seconds to the boiling aqua-regia solution above described. This developed a better crystalline structure, though not a thoroughly satisfactory one. The same micro-section of platinum was therefore again exposed to the action of boiling aqua regia for a further period of 45 seconds, resulting in the development of a beautiful crystalline structure which manifested not only the large or primary crystal grains but also the secondary or very minute crystalline development which is illustrated in the following photographs:

Fig. 1 shows the micro-crystalline structure of pure platinum

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\* *Proceedings of the Royal Society*, Vol. LXIX.



as seen in transverse section by oblique illumination at a magnification of 50 diameters.

Fig. 2 shows, at a magnification of 120 diameters, the crys-

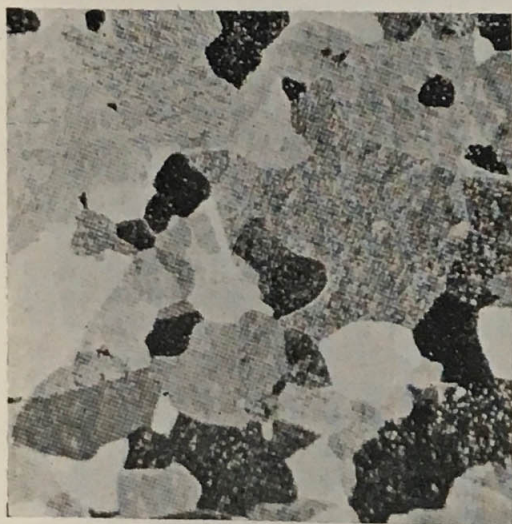


Fig. 1. Transverse section, oblique illumination.  
Magnified 50 diameters.

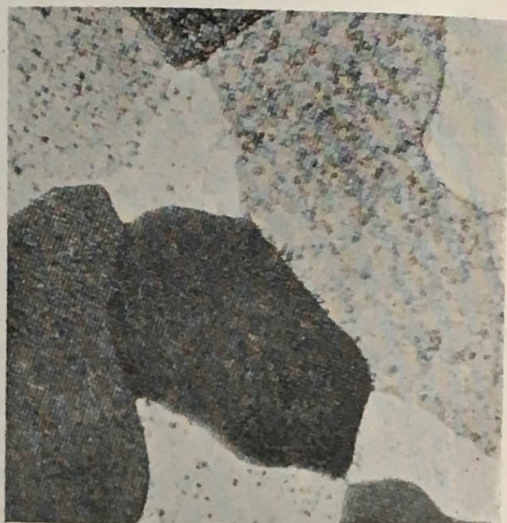


Fig. 2. Transverse section, vertical illumination.  
Magnified 120 diameters.

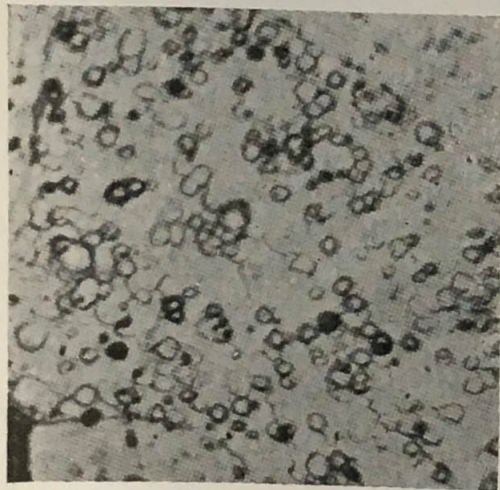


Fig. 3 and 4 primary and secondary crystals. Vertical illumination.  
Magnified 360 diameters.

talline structure as indicated in transverse section by the direct or vertical method of illumination. This micro-section illustrates the general form of the primary or large crystal grains, and also

indicates the existence of very minute crystals within the boundary lines of the larger crystal grains.

The structure and general orientation of the minute secondary crystals, as seen in section by vertical illumination at the higher magnification of 360 diameters, are illustrated in Figs. 3 and 4.

The larger or primary crystal grains were observed to consist of irregular polygons of varying size, the etched indications of the facet junctions being often clearly and sharply defined.

The minute or secondary crystals (whose inter-crystalline junctions were also clearly seen) were in the mass observed to be in varied positions of orientation, but the orientation was generally identical, or on the same plane, within the area of each larger crystal grain.

The general orientation of the smaller crystals varied, however, in each separate larger crystal, and the consequent varied reflection of the light has given the face of the micro-section, as a whole, the appearance of lighter or darker areas in the photomicrographs. This effect is merely optical.

In some portions of the mass there were observed minute triangular crystals; these appear, however, to be only developments resulting from the cutting of certain crystals in section.

The general micro-crystalline structure of platinum was observed to be allotriomorphic in character and derived from a system of interfering cubes and octahedra, the cubic and hexagonal form being frequently noticeable.

The size of the large crystal grains varied from about .002 inch to .04 inch in size, and the smaller crystals ranged from about .0002 inch to about .007 inch.

There were indications that the smaller or secondary crystals were each built up of even more minute crystalline ramifications.

The crystalline structure of platinum appears generally to resemble that of gold and silver.

The descriptive words "primary" and "secondary" crystals are only used in relation to size, and they are not intended to convey the idea of distinctive times of formation during solidification.



## ADDENDUM

In connection with the present investigation reference may be made to the valuable researches of Professor Liversidge on "The Crystalline Structure of Gold and Platinum Nuggets."\* On comparison there appears to be considerable identity between the crystalline structure of natural platinum nuggets and that of the platinum ingot examined by the author, the crystalline jointings appearing to be similar in character.

A general analogy appears also to exist between the crystalline structure of pure platinum and that of pure gold, which is noticeable on comparing the illustrations in the present paper with those in the following papers, viz.: "On the Structure of Gold and Gold Alloys," by Professor J. O. Arnold,† and "On the Structure of Metals, its Origin and Changes," by M. Osmond and Sir Roberts-Austen.‡

This identity of structure is further seen on referring to the illustrations in the paper on "The Microscopic Structure of Gold and Gold Alloys," by Mr. Thomas Andrews.§

## MICROSCOPIC EFFECTS OF STRESS ON PLATINUM ||

By THOMAS ANDREWS and CHARLES REGINALD ANDREWS

THE microscopic effects of stress on platinum do not appear to have been studied. An ingot of pure platinum was therefore prepared, and from this a portion was accurately machined in the form of a cube, 0.30 inch square, which was afterwards carefully microscopically polished, and then subjected to compressive stress in the testing machine.

Prior to the application of stress, and for comparative purposes, a polished face of the platinum cube was microscopically

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\* *Journal, Royal Society of New South Wales*, 1897, Vol. XXXI, p. 70, Plate 9.

† *Engineering*, Vol. LXI, 1896, p. 176.

‡ *Phil. Trans., A*, 1896, p. 417, Fig. 10, Plate 9, and Fig. 16, Plate 10.

§ *Engineering*, September 30, October 28, December 9, 1898; *The Metallographist*, Vol. II, p. 105.

|| Read before the Royal Society, May 15, 1902.

examined, but an even polished surface only was observed. A force producing a compression of 10 per cent on the total height of the cube was then applied, and microscopic observations were taken at high magnifications of the effects of the stress on the micro-crystalline structure of the platinum cube. The particulars of the stress applied are given on Table I.

The polished side of the cube upon which the high-power microscopic examination was made was the one in line, or in parallel, with the direction of the compressive force. The results observed were very interesting, and confirm Professor Ewing's and Mr. Rosenhain's observations on the peculiar manner in which the crystalline structure of a mass of metal becomes altered when under stress.\*

On comparing the appearance of the platinum after having been stressed with its appearance before the application of stress, it was found that many of the individual large or primary crystal grains forming the mass had, under the influence of the strain, developed innumerable fine "stress bands" or "slip bands," or indications of crystalline slip. The main lines of disruption were noticed in many instances to roughly approximate to an angle of about  $45^\circ$  to the line of the compressive force on the crystal sectional facets. Between these large indications of crystalline slip were observed a number of extremely fine lines, indicative of the nature of the crystalline slip; many of these ran more or less in parallel lines in each individual primary crystal or crystal grain, the direction of the orientation (or line of the normal cleavage) of each primary crystal grain apparently influencing the direction of the "slip bands," as seen in section.

Owing to the varied orientation of the different crystals in the mass of the platinum,† the lines of cleavage as indicated by the minute "slip bands," were often seen at varied angles to the line of the straining force.

These experiments have also confirmed the observations of

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\* Bakerian Lecture, 1899, *Phil. Trans., A*, Vol. CXCI, 1900, p. 353.

† See "Micro-crystalline Structure of Platinum," by Thomas Andrews, *Roy. Soc. Proc.*, Vol. LXIX, March 21, 1902, No. 457, p. 433; also "Microscopic Structure of Gold and Gold Alloys," by Thomas Andrews, *Engineering*, September 30, October 28, December 9, 1898; also "Micro-metallography of Iron," by Thomas Andrews, *Roy. Soc. Proc.*, Vol. LVIII, 1895.



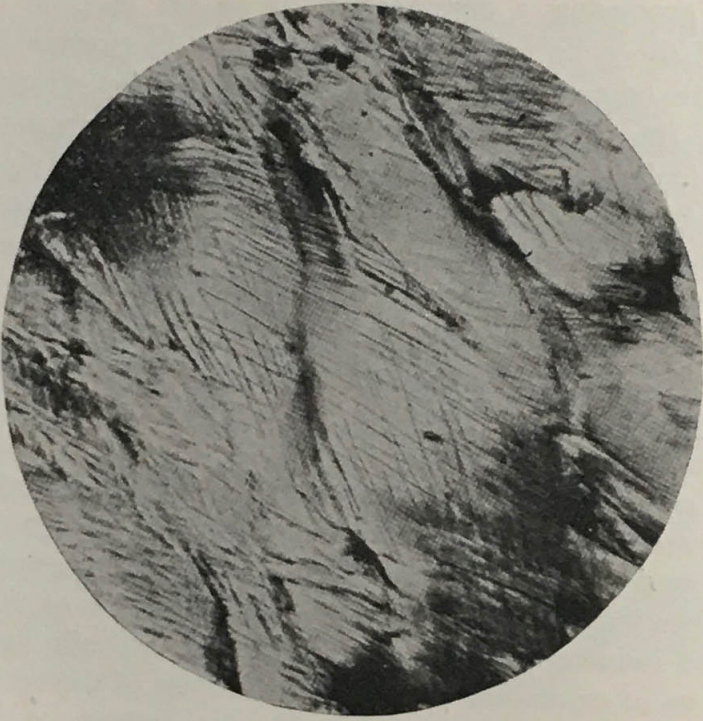


Fig. 1. Structure of platinum after compression, showing crystalline slips. Magnified 120 diameters

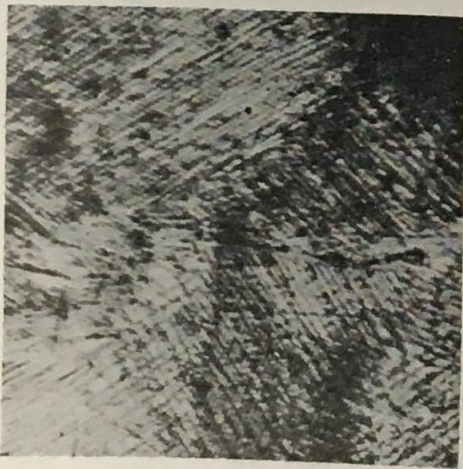


Fig. 2. Crystalline slips. Magnified 120 diameters.



Fig. 3. Same as Fig. 2. Magnified 250 diameters.

Professor Ewing and others, that stress alone, without etching, sometimes renders manifest the lines of intercrystalline junction of the large or primary crystal grains of a stressed metal, providing that the stress is of sufficient intensity. This will be seen on reference to the accompanying illustrations, Figs. 1, 2 and 3.

The general appearance of the disintegration of the large or primary crystal grains, produced by the pressure, on the pure platinum cube, was the apparent breaking up of the crystalline structure of the metallic mass, as seen in section, roughly diagonally to the line of the compressive force. The area enclosed by the main lines of disruption roughly approximating to the size of the large primary crystal grains. The distances between the extremely fine lines, or "slip bands," appeared roughly to coincide proportionately with the size of the secondary or most minute crystals forming the mass, the finer "slip bands" appearing to indicate the crystalline slip which had taken place along the facets of the smaller or secondary crystals. The direction, however, of the main lines of the crystalline disruption did not appear always to coincide with the intercrystalline facet junctions of the large or primary crystal grains. The lines of least resistance, or greatest crystalline slip, seemed chiefly to develop at an approximate angle of about 45 degrees to the pressure line, as previously mentioned; but the line of greatest weakness in the mass structure of the metal was not always at that angle with the line of the disruptive force.

The previous description generally and approximately indicates the effect observed, but the breaking up of the crystalline structure of the metal appeared to be subject to modifications according to varied internal or external conditions.

The authors hope that these experiments may prove of use in affording an indication of the comparative behavior of this "*ἀριστο-κρατία*" or noblest metal platinum, with the behavior of the constructive metals, copper, nickel, iron and steel, when under the influence of stress; and the experiments have also shown that the microscopic influences of stress in the heavy metal platinum are analogous to those which have been observed in metals of lower specific gravity.



*Compressive Stress applied to Platinum Cube.*

	Cross-section Dimensions, inches	Area Square inches	Height before test Inches	Stress required to compress sample 10 per cent of its original height Tons per square inch.
Platinum cube	0.30 × 0.29	0.087	0.30	12.82

**THE MICROSCOPE AND THE METALLURGY OF STEEL\***

By C. H.

IT is not the intention of this article to explore all the details in connection with the practical application of the art of microscopy to the study of the physical construction of steel. A brief review in a general way will be given on the microscopy of cast steel as the result of a study and a long series of experiments conducted by the Leighton & Howard Steel Company, formerly the Shickle, Harrison & Howard Company, of St. Louis.

Cast steel, by its great strength, its marvelous ductility, its shock-resisting qualities and its adaptability to many purposes where such qualities are necessary, is a material worthy of the admiration of engineers. The study to improve upon the manufacture of and the qualities of cast steel offers an inviting field for research and investigation. When cast-steel is used it is but fair to both manufacturer and purchaser that it should meet the expectation of all concerned. To make cast-steel that is a credit to the manufacturer and satisfactory to the consumer is an art that is making rapid progress. It has been evident to engineers and metallurgists for some years that to explain some of the physical properties of cast steel, a knowledge beyond that gained by a mere chemical analysis was needed. It was when this point was fully realized that the microscope came to the rescue.

\* *The Railroad Gazette*, June 13, 1902. This article and illustrations are reproduced here through the courtesy of the *Railroad Gazette*.

By its aid the discovery was made that only by a certain crystalline arrangement was steel, of a known chemical analysis, and for a specific purpose, in the best possible physical condition to perform successfully the service demanded. Chemistry cannot explain the different degrees of crystallization. By chemical analysis we learn the ultimate composition. By the microscope can be acquired, in a great measure, information regarding the structure of steel.

Of great importance in fixing the proper crystalline structure is an intelligent knowledge of heat treatment and thermal conditions throughout. The accompanying engravings from photographs made through a microscope, show plainly the changes in the structure of cast steel as the result of annealing. The photographs were made from specimens from the same piece of steel, subjected to different temperatures. Fig. 1 shows the usual structure of normal steel as cast. The light parts are known as "ferrite" or carbonless iron. The dark parts are known as "pearlite," an alloy of iron and carbon. Steel with this structure is brittle, stands a poor bending test and does not give good results in ductility; nor does it well resist shocks or torsional and transverse strains.

Fig. 2 shows a structure produced by judicious heat-treatment, or annealing. The ultimate composition and the proportion of pearlite and ferrite are the same. The difference is in the diffusion of the two constituents, the photograph showing a fine micro-grain and a complete obliteration of the coarse crystals. Such steel is capable of resisting all strains without manifesting brittleness. It has great ductility and can be bent flat upon itself without fracture.

Fig. 3 shows a structure formed by imperfect or over-heating. It will be seen there is a tendency toward a growth of a larger micro-grain, along with an enlargement of the crystals of the mass. Such steel is less brittle than the first specimen and falls short of the ductility and toughness of the second.

Fig. 4\* presents a more extensive exhibit of results from a series of these experiments and shows clearly the effect of heat on the physical properties of the steel. All of these specimens were from one piece of steel, having 0.39 per cent of

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\* Fig. 4 was published in the January, 1902, number of *The Metallurgist*, page 76.



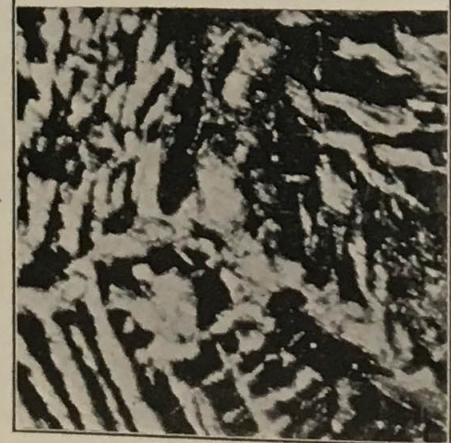


Fig. 1. Cast steel.

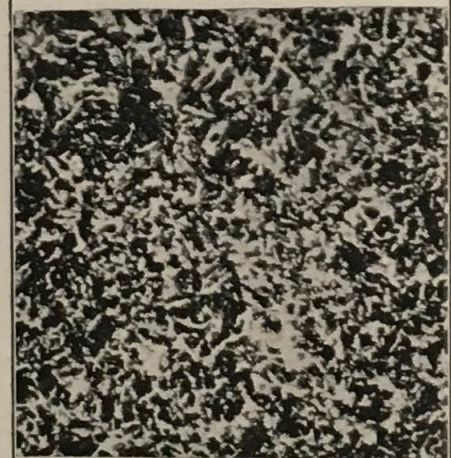


Fig. 2. Cast steel properly annealed.

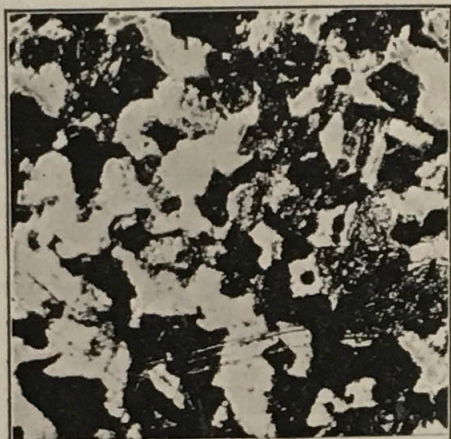


Fig. 3. Cast steel over-annealed.

carbon. The treatment was applied in an electrical furnace and the temperature taken with a Le Chatelier pyrometer. The pieces were cooled slowly and bent when cold. Fig. 5 shows a specimen of properly annealed steel before and after subjection to a tensile test. The tensile strength developed was 69,600 lbs., with an elongation of 32 per cent and a reduction in area of 42.18 per cent.

To one trained in metallography a glance is sufficient to determine by the microscope the heat treatment to which a given piece of cast-steel has been subjected. The size, shape and arrangement of its crystals and microstructure are all controlled in a finished casting by heating and cooling slowly or quickly.

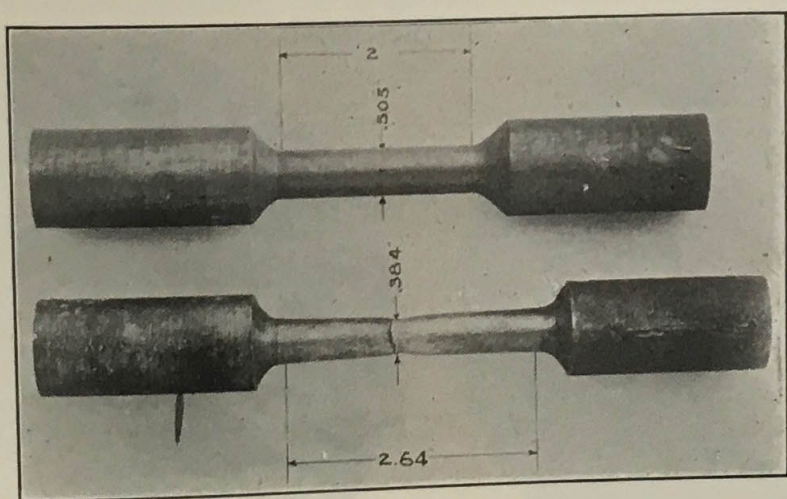


Fig. 4. Specimen of properly annealed steel before and after testing.

The physical properties of the metal are as closely allied to the structure as the structure is to the heat-treatment. To produce desirable results in cast-steel it is necessary to know what thermal conditions to observe in order to throw all possible safeguards around the product. This knowledge can only be gained by scientific pyrometry and comparative study of microstructures as affected by heat treatment, together with the known chemical composition.

It is the aim of the metallurgist to apply to industrial problems deductions gained by investigation and research conducted in the laboratory. The laws producing certain effects are observed by experiment, then with the needful instruments the practical operation of annealing castings is much simplified,



and all notions so diversified on that question can be lifted out of guesswork to the plane of definite information. With a pyrometer of reliable accuracy, and the microscopical tests to verify the temperature compared with established standards prepared in the laboratory, the industrial operation of annealing becomes a laboratory practice.

A study of the various structures with known thermal conditions impresses one with their striking beauty and the immense value of the information derived therefrom. With a known chemical composition and a known history of heat treatment, it is possible to predict with an element of success the physical qualities of steel castings when examined with a microscope; hence its value to the metallurgist or engineer is of such importance that it becomes indispensable.

## METALLOGRAPHIC NOTES

**The Structure of Steel Rails.**—The accompanying photomicrographs were taken at the works of the Maryland Steel Co. and sent to us by Mr. S. S. Martin, superintendent of the steel department. They illustrate in a striking manner the influence of the sawing operation upon the structure of the metal in immediate contact with the saw.

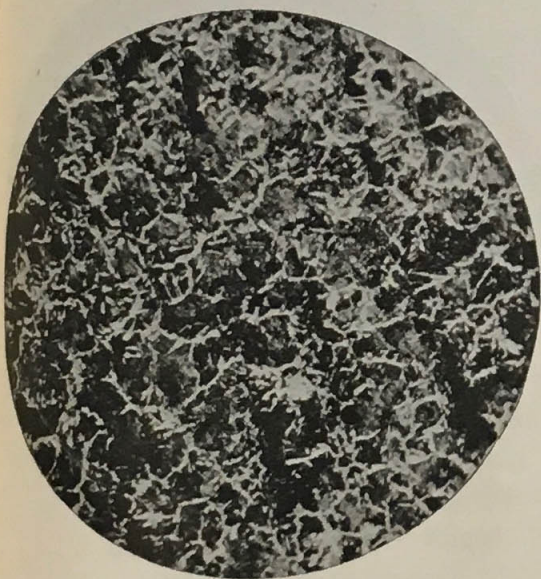


Fig. 1.

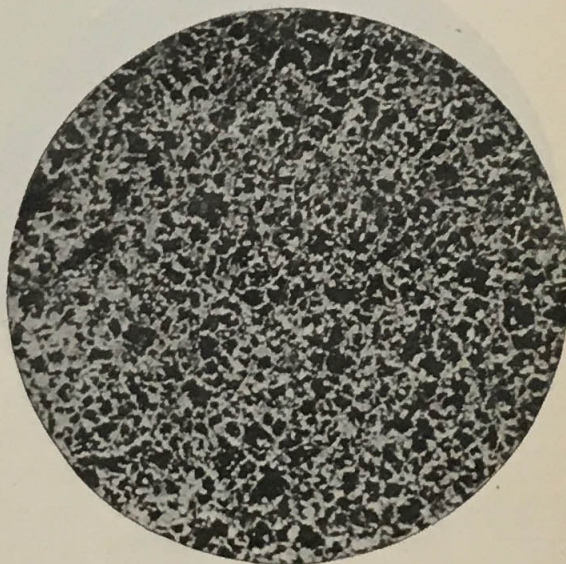


Fig. 2.

Fig. 1 shows the normal structure of a rail in the center of the head, and a considerable distance from the sawed end. This rail was hot sawed as usual, to proper lengths, and then a half inch section was cut from the hot sawed end by means of a slow speed *cold saw*. In this way a section was obtained which had been hot sawed on one side and cold sawed on the other. Fig. 2 exhibits the structure of the metal on the hot sawed side, and Fig. 3 on the cold sawed. The hot sawed has a much finer structure than the normal structure of the rail (Fig. 1), while the cold



sawed surface is, if anything, coarser. Upon removing 0.07 inch of metal from both sides the structures shown in Figs. 4 and 5 were obtained, which do not differ materially from the normal structure of the rail (Fig. 1). The effect of the temperature at which sawing is done affects only, therefore, the structure of the metal to a very slight depth.

The very great difference between the structure of the hot sawed and of the cold sawed sides can readily be accounted for. Upon leaving the finishing rolls the temperature of the rail (at least in the center of the head) was still well above the critical

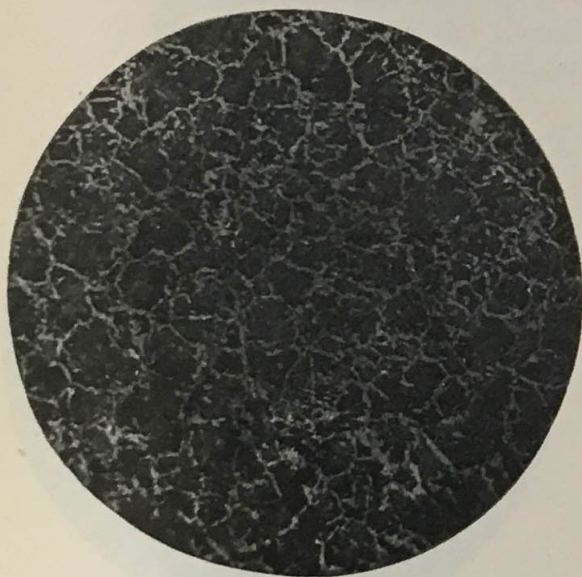


Fig. 3.

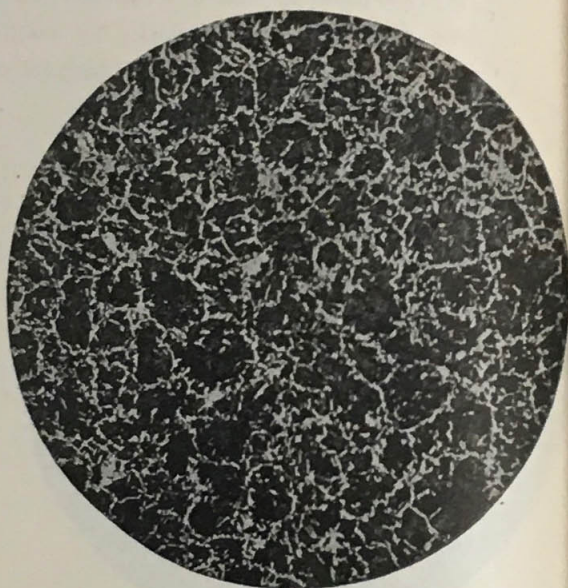


Fig. 4.

point, i. e., the rail was still in the crystallizing range (which extends from the point of solidification to the thermal critical point). On slow cooling the metal crystallized, resulting in the structure of Fig. 1. The hot sawing of the rail means that the metal which is in close vicinity to the saw is not allowed to cool *undisturbedly*, and therefore to crystallize; the particles are kept in motion, the *hot work is continued* until a temperature below the critical point is reached, at which there is no further growth of crystal. The structure of the hot sawed side is, therefore, a fine structure such as is exhibited in Fig. 2. It is a striking illustration of the bene-

ficial effect of hot work upon the structure and of the importance of low finishing temperatures. Could one produce a rail showing a structure like that of Fig. 2, in the center of the head, and throughout the entire length, such rail would combine great ductility with excellent wearing quality. It also follows from this

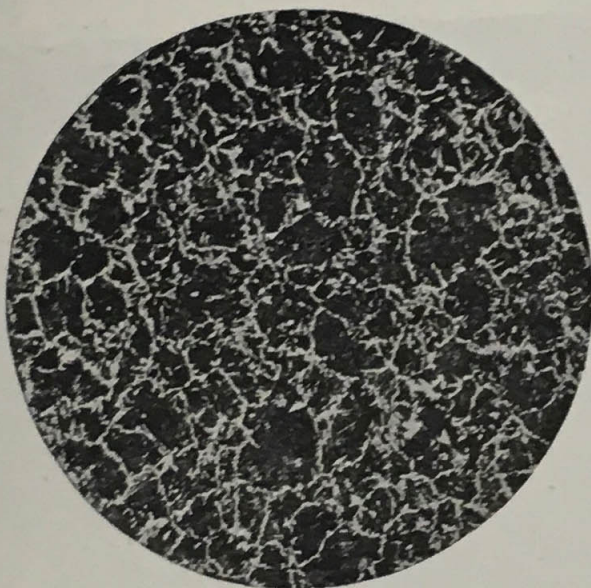


Fig. 5.

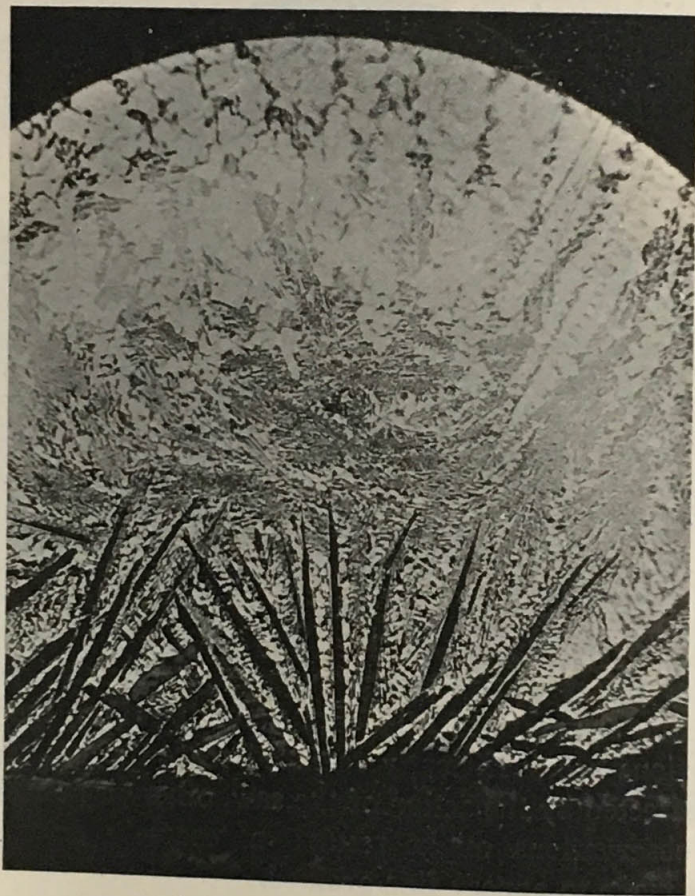
significant effect of hot sawing upon the structure, that when studying the structure of rails care should be taken that the polished sections are sufficiently removed from the hot sawed side to be unaffected by the action of the saw, otherwise seriously misleading observations might result; a coarsely crystalline rail might be made to appear fine-grained.

**Structure of Copper and Antimony Alloys.**—The accompanying photomicrograph taken by Mr. J. E. Stead illustrates forcibly the value of the "superposing" method for the preparation of alloys to be studied under the microscope. The method consists of first melting the metal of highest specific gravity and then pouring on top of this, in a molten condition, the other, lighter metal. The two metals will alloy in such a way that a vertical cross-section will show crystals of a pure metal at one end and crystals of the other metal at the other end, while between these the metals will be found alloyed in all proportions. It will



be readily seen that such method will make the preparation of a number of alloys containing various proportions of the constituent metals unnecessary. It was described by H. Le Chatelier (*The Metallographist*, Vol. IV, page 17), but has also been worked independently by Mr. Stead.

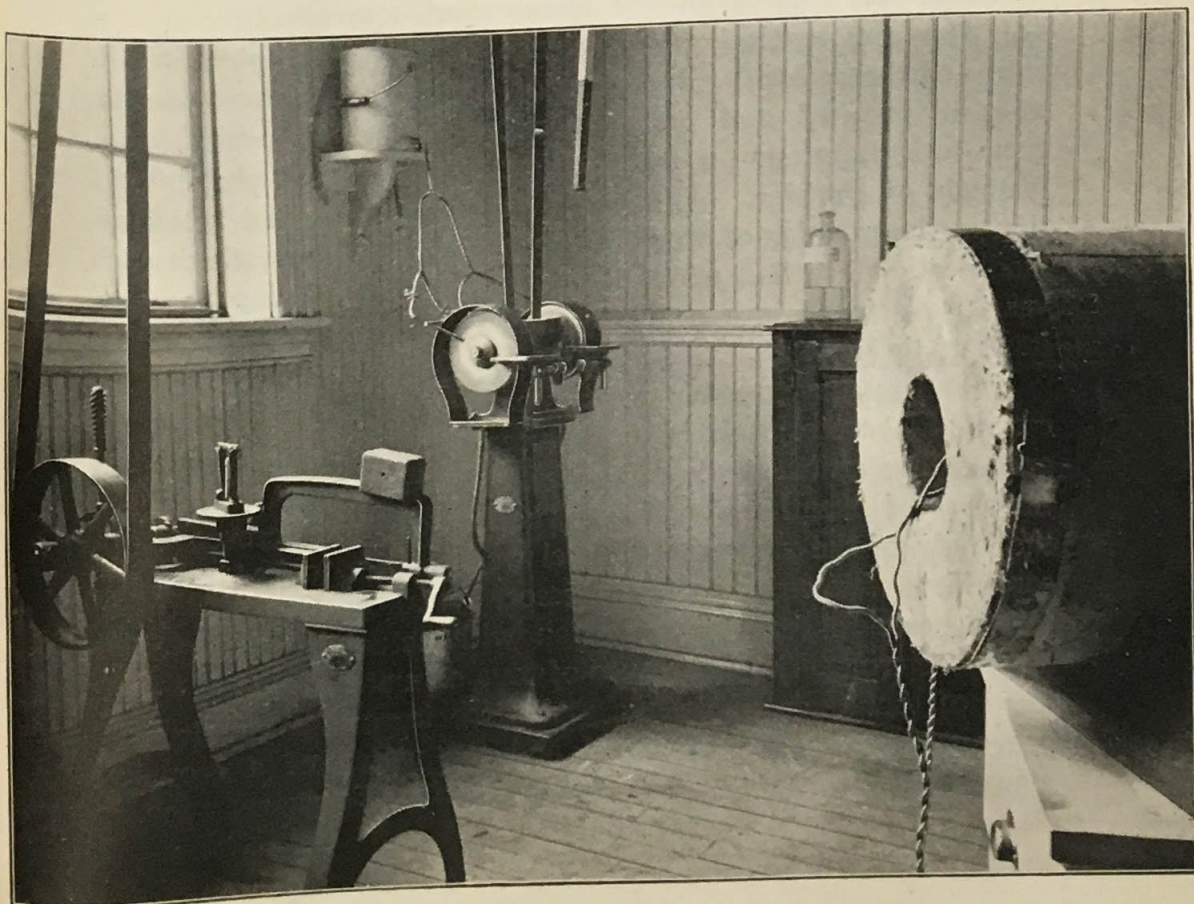
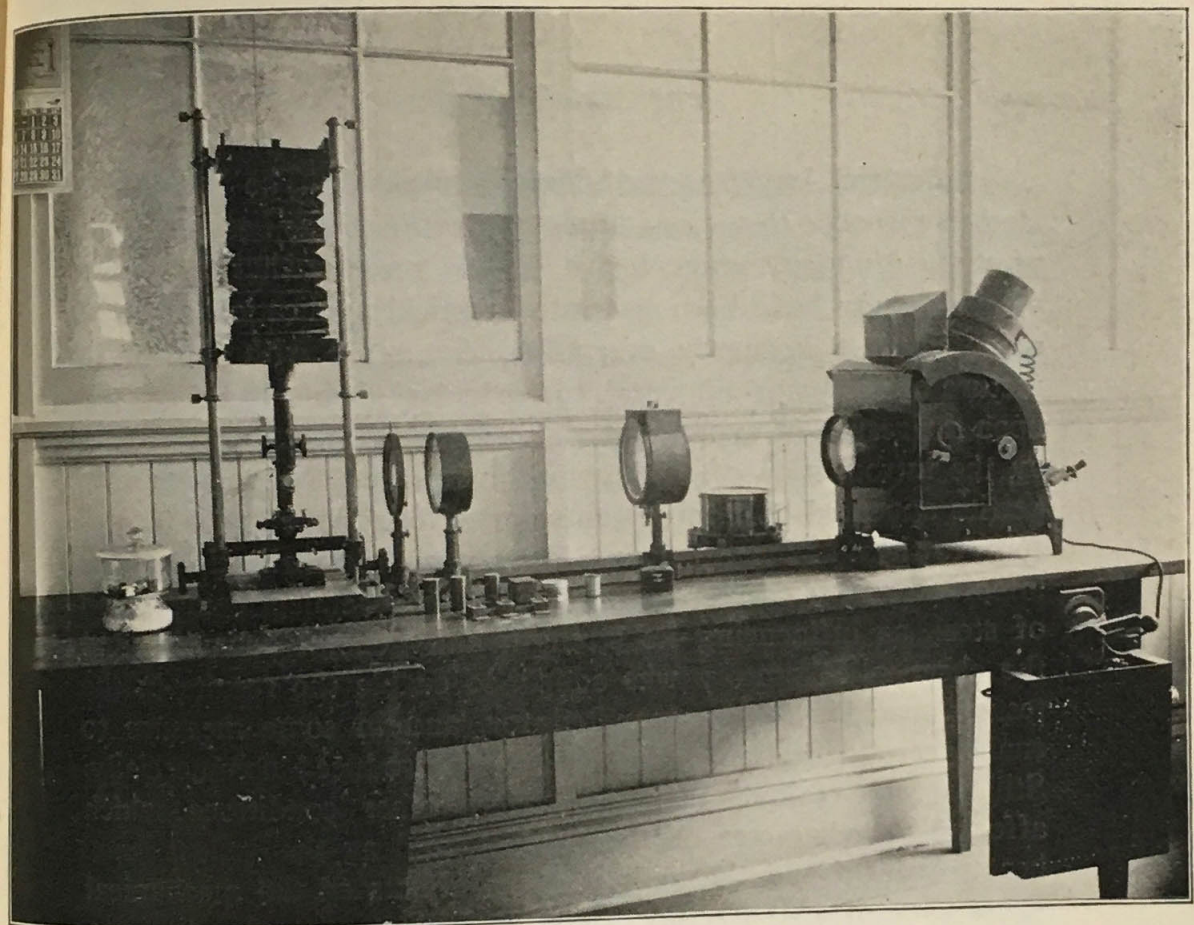
In the accompanying illustration the white trains of crystals



at the top are antimony, the central portion the eutectic alloy of Sb and  $\text{SbCu}_2$ , and the wedge-shaped crystallites at the bottom  $\text{SbCu}_2$ .

**Metallography at the Westinghouse Machine Co.**—The illustrations on the opposite page show the very complete metallographic equipment of the Westinghouse Machine Co. of Pittsburgh, Pa. It includes the following parts: Power hack saw, polishing machine, electric arc lamp with optical bends and rheostat, special microscopic and accessories, photomicrographic camera, electric furnace and Le Chatelier pyrometer.





Metallographic equipment of the Westinghouse Machine Co. — Pittsburg, Pa.



Manager to Messrs. George Cradock & Co. and communicated, in conjunction with Mr. Stead, a paper on steel for wire-making to the Iron and Steel Institute last year.\*

**Metallography at the Technical Schools.**—*Massachusetts Institute of Technology.* The importance of metallography, the microscopical study of the structure of metals, is being more and more recognized. Even the iron foundries are now using this means for controlling the quality of their materials, in order to produce even and reliable results. The course in this subject has been transferred from the Chemical to the Metallurgical Department, where it naturally belongs. Lectures and laboratory work in this line have been added to the curriculum for all regular students in the Course in Mining Engineering and Metallurgy, and advanced work is provided also for those who elect this subject for a thesis or wish to work upon it with the view to obtaining an advanced degree. This work is under the charge of Professors Hofman and Fay.—*Extract from the Annual Report of the President and Treasurer.*

**Other Scientific Schools.**—The following schools have recently added to their laboratory equipment the necessary apparatus to carry on metallographic work. McGill University, Montreal, Canada, Michigan College of Mines, Houghton, Mich., and Worcester Polytechnic Institute, Worcester, Mass.

**Metallography and Industrial Firms.**—The following firms and individuals have since the first of the year installed metallographic outfits in their laboratories or have added to the equipment they already had: National Enameling and Stamping Co., East St. Louis, Ill.; Trenton Iron Co., Trenton, N. J.; Ajax Metal Co., Philadelphia; William Cramp & Son, Ship and Engine Co., Philadelphia, Pa.; Cambria Steel Co., Johnstown, Pa.; National Tube Co., McKeesport, Pa.; General Electric Co., Lynn, Mass.; Watertown Arsenal, Watertown, Mass.; Enrique Touceda, Albany, N. Y.; Crucible Steel Co. of America; United Engineering Foundry Co., Pittsburg, Pa.; American Steel Foundry Co., Granite City, Ill.

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\* It is a significant fact that in the majority of cases metallographic researches are being carried on by the successful candidates for this scholarship.—ED.

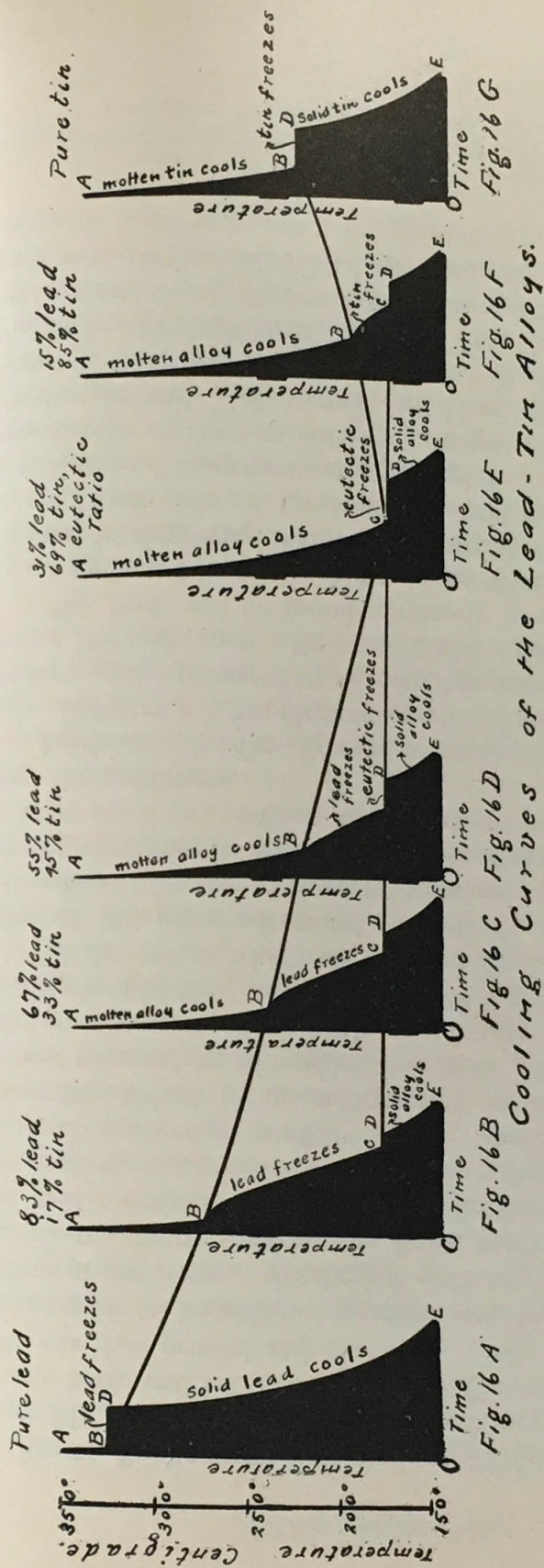
**Recent Publications.** — *Metallurgical Laboratory Notes*, by Henry M. Howe. In our January issue we reviewed Professor Howe's book entitled "*Metallurgical Laboratory Notes*." The book was then printed and practically ready for the binder, and we expected to be able to fill orders within a week after the writing of our notice. At the last moment, however, the author decided to greatly enlarge the book, more than doubling its size, and requested us to delay publication accordingly. The new experiments which Professor Howe desired to describe had all to be conducted carefully in the laboratories of Columbia University before being incorporated in the book. This, of course, required considerable time and will explain the long delay in issuing the book. We now expect to have it ready for delivery within four weeks and possibly earlier.

All the orders which we have received to this date will be filled at the price advertised, namely \$1.25, or \$1.10 to our subscribers. Owing to the late addition to the book, however, greatly increasing its size, we are compelled to advance the price to \$2.50 per copy, or \$2.25 to subscribers when ordered directly from the publishers.

*Jahrbuch für das Eisenhüttenwesen.* (Yearbook of the Iron Industry.) Supplementary to *Stahl und Eisen*. An account of the progress in all departments of the Iron Industry during the year 1900. By Otto Vogel. Düsseldorf, 1902.

This book will be found extremely interesting and valuable to all steel metallurgists able to read German. The most important developments that have occurred during the year 1900 in all departments of the iron and steel industry are concisely described and numerous references given. The book is divided into fifteen sections as follows: General Considerations, Fuels, Production and Measurement of Heat, Refractory Materials, Slags, Ores, Iron and Steel Plants, Production of Pig Iron, Foundries, Production of Malleable Iron (and Steel), Working of Iron and Steel, Properties of Iron and Steel, Testing. Each section is further subdivided as required.





The illustration reproduced here is taken from Professor Howe's book on Alloys, soon to be published. It illustrates in a most satisfactory manner the construction of the solubility curves of metallic alloys. Independent cooling curves of pure lead, pure tin and of five alloys of lead and tin were constructed in the usual way and wooden models of these made and photographed as shown in the illustration. All the points marking the beginning of the solidification of each metal or alloy were then united, as well as the horizontal parts of the models which correspond to the solidification of the eutectic.







GEORGE CHARPY

# The Metallographist

A QUARTERLY PUBLICATION DEVOTED TO THE STUDY OF METALS, WITH  
SPECIAL REFERENCE TO THEIR PHYSICS AND MICROSTRUCTURE,  
THEIR INDUSTRIAL TREATMENT AND APPLICATIONS.

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Vol. V

OCTOBER 1902

No. 4

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## GEORGE CHARPY

GEORGE CHARPY was born in Lyons in September, 1865. From 1885 to 1887 he studied at the École Polytechnique of Paris, and from 1887 to 1892 he remained connected with that Institution as assistant in physics and chemistry. During these years devoted to teaching, he published, in collaboration with Mr. Gautier, a book entitled "Leçons de Chimie," of which three editions have been issued. While thus engaged Mr. Charpy also wrote a number of papers on physical chemistry, and in 1892 he presented his memoir on "Researches on Saline Solutions," to the Faculty of Science of Paris for which he was awarded the degree of doctor of science.

In 1892, Mr. Charpy was appointed chemist of the Central Laboratory of the Navy, at Paris, and soon after began his well-known metallographic investigations. He published a very important paper on the hardening of steel for which he received a prize from the Société d'Encouragement pour l'Industrie Nationale.

In 1898 Mr. Charpy left the laboratory of the Navy to become Chief Engineer of the Compagnie des forges de Chatillon, Commentry et Neuves Maisons at Montluçon, a position which he still holds. For his metallurgical exhibit at the Paris Exposition of 1900, he received a gold medal. The readers of this



journal are well acquainted with his contributions to metallography. They have been many and most valuable. Mr. Charpy's researches testify to his thorough scientific training and great experimental skill; his writings are characterized by extreme lucidity and a constant desire to extract from his experiments deductions of industrial value. In the light of what he has already accomplished at so early a period of his life, we are justified in looking to him for many additional contributions to the advancement of metallurgy and metallography.

His most important papers dealing with metallography and related subjects are given below in chronological order:

Researches on Saline Solution. Thesis presented to the Faculty of Science. Paris, 1892.

Note on the Hardening of Steel. *Bulletin de la Société d'Encouragement pour l'Industrie Nationale*.

Researches on Alloys of Copper and Zinc. *Société d'Encouragement*, 1897.

Microscopic Study of Metallic Alloys. *Société d'Encouragement*, March, 1897. *The Metallographist*, Vol. I (1898).

Study of White Alloys called Antifriction. *Société d'Encouragement*, June, 1898. *The Metallographist*, Vol. II (1899).

Study of the Tenacity of Alloys at High Temperatures. *Société d'Encouragement*, February, 1899.

The Equilibrium of Iron-Carbon System (with L. Grenet). *Société d'Encouragement*, March, 1902. *The Metallographist*, Vol. V (1902).

For his important investigations described in the above papers he was awarded a prize by the Mechanical Section of the Académie des Sciences of Paris.

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## THE INTERNAL STRUCTURE OF IRON AND STEEL WITH SPECIAL REFERENCE TO DEFECTIVE MATERIAL\*

By S. A. HOUGHTON

IN the first place I would call attention to the title of this lecture — "The Internal Structure of Iron and Steel." I had at first intended to have described it as the *Microstructure* of Iron and Steel, which is indeed the usual method of describing the structure of the metals, owing to the fact that a microscope

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\* *Proceedings Institute of Marine Engineers*, April, 1902.

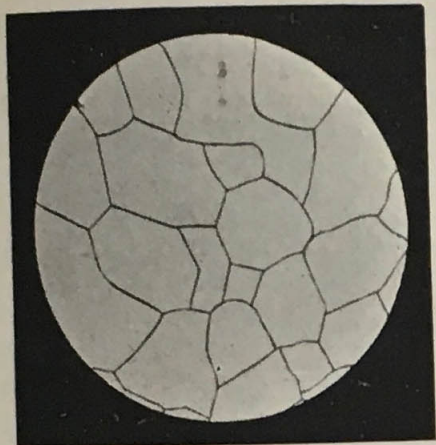
is generally required. It occurred to me, however, that some of you might imagine that there was a difference between the microstructure and the ordinary structure. There is none — there is only one structure of metals, and although as a rule it is necessary to use a microscope to examine it properly this is not invariably the case, and in fact I shall be able to show you a photograph of some iron, the general structure of which is quite visible to the naked eye. That there is some ground that a misapprehension of this nature may occur is, I think, shown by the history of this method of investigating the characteristics of metals. It is now nearly forty years since it was originated; the exact date can be stated with certainty, as, unlike many other discoveries, there is no doubt as to whom the honor is due for first using this method of examination. The first, and for a long time the only, person to do so was an Englishman — Dr. Henry Clifton Sorby, of Sheffield, who is, I believe, still alive. Dr. Sorby is a man of science and has made several discoveries and inventions, but he was not directly connected with the iron industry; indeed, he was at the time mentioned investigating the characteristics of igneous rocks, and it was in order to obtain information respecting certain of their peculiarities that he turned to examine iron, using the same methods as in petrology, i.e., he cut and polished sections so as to be quite free from scratches, afterwards etching them if necessary with acid so as to define the constituents more clearly. (I may say that I have had the pleasure of seeing some of Dr. Sorby's sections, and although the time of preparation has now been much shortened yet in respect to finish we have not appreciably advanced.) The same procedure is still employed, and the keynote of the whole science is, to use Dr. Sorby's words, that steel must be regarded as an artificial crystallized igneous rock, and to get a complete knowledge of it must be examined as such.

Well, although Dr. Sorby read several papers about 1864, yet they excited little attention amongst those engaged in the iron and steel industry, the reason being probably that the practical men of that day considered that looking at metals through a microscope only showed a microscopic structure as different to the ordinary structure, and that it was too ultra-scientific to be of any use; they had heretofore to rely entirely on their chemists. Now chemistry is a great and very useful science, and I do not

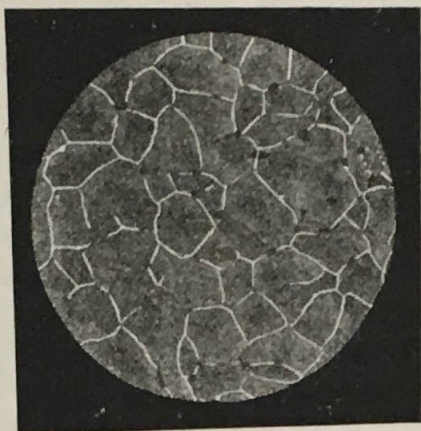


wish to say anything against it, but, as chemists themselves will tell you, the characteristics of metallic alloys have been and are quite unexplainable by their science. Let me take an example: by the addition of very small quantities of bismuth to copper, that metal, usually so ductile, becomes quite brittle, and, moreover, the electrical conductivity decreases. Now there is no relation between the rate at which these alterations take place and the atomic weight of bismuth, i.e., the decadence is gradual and not in steps. The reason for these peculiarities remained a mystery until the alloys were examined under the microscope by Professor Arnold, of Sheffield University, and the slides now shown are copied from some micrographs published by him. In slide No. 1a is seen a section of pure copper which consists of a number of homogeneous crystals; slide No. 2a is that of copper containing .5 per cent of bismuth. It will be seen that the crystals are bordered by white lines, and there are also a number of dark spots, the latter containing nearly pure bismuth. The nature of the boundaries of the crystals is clearly shown in the next slide (No. 3a), from which it will be observed that each crystal is bounded by a band of what is apparently an alloy of bismuth and copper, and also that there is a distinct line between each which is, according to Professor Arnold, a line of cleavage. It will at once be seen why the strength of the metal has so diminished, and also why the electrical resistance has become greater, the current now having an increased resistance to overcome in passing from one crystal to another. You will see from this illustration how helpless pure chemistry is for solving many of the problems which confront the metal worker. Nevertheless for about twenty years after Dr. Sorby began his work it was practically the only scientific method adopted, and even to-day there are many people who attach more importance to a chemical analysis than to an examination of the actual structure of a metal. Before leaving this question I desire to draw your attention to a very important weak point in most analyses, i.e., they give the quantities of certain elements, but take no notice of others that may be present in even large quantities; in fact, the chemist is unable as a rule to say whether an element is present unless he specially analyzes for it.

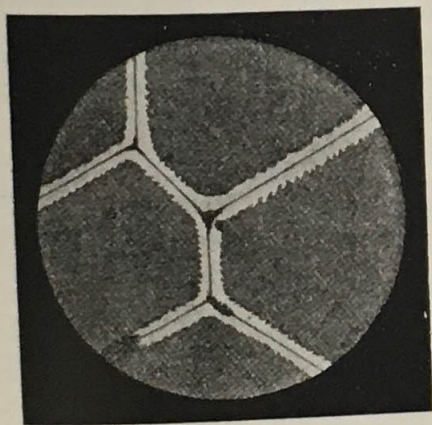
The result of sole reliance on this science has been to materially delay the progress of steel manufacture, and has been



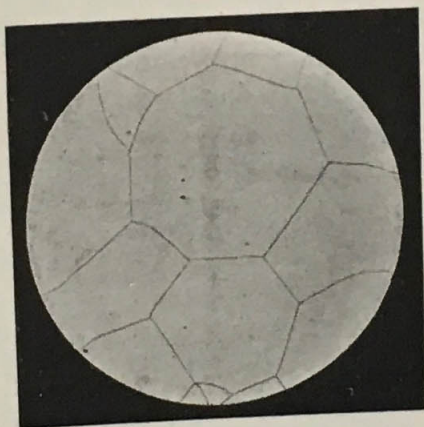
Slide No. 1a. — Pure Copper,  
8 diam.



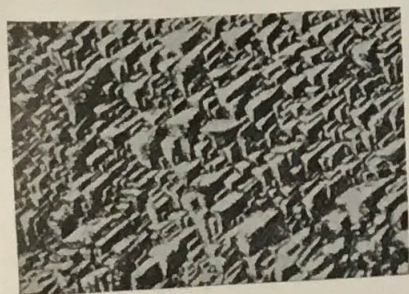
Slide No. 2a. — Copper containing  
.5 per cent bismuth, 8 diam.



Slide No. 3a. — Copper containing  
.5 per cent bismuth, 48 diam.



Slide No. 4a. — Pure Swedish  
Iron.



Slide No. 5a. — Silicon Steel,  
60 diam.



Slide No. 6a. — Silicon Steel,  
60 diam.



indirectly the cause of much wasted material and mysterious failures. When these latter were investigated the steel maker would triumphantly produce a piece of paper on which was written that there was so much carbon, manganese, phosphorus or sulphur, all of which were within the usually accepted limits, and, although perhaps he did not directly say so, he would lead one to infer that the cause of the failure must therefore be attributed (to use the words of the old charters) "to the act of God or the Queen's enemies." This of course was not very satisfactory; unfortunately such failures still occur, and although the number is very small compared with the amount of steel made, yet so long as the causes cannot be ascertained and remedies suggested the matter is to my mind a very serious one, and every possible method of investigation should be tried to clear it up.

It must also be borne in mind that, as Professor Arnold has stated, we have got to face the fact that steel which has stood the ordinary tests satisfactorily may yet fail under normal treatment, and that on many such failures chemical analysis throws no light whatever.

As previously mentioned, the method which I am bringing to your notice to-night is that which shows the structure of the material, and I think you will all agree that from the most casual consideration it is one which deserves a very high place in any investigation. It is indeed curious to think that people have been content for so long to manufacture and use in such quantities a material such as steel of whose structure they were entirely ignorant. Indeed even quite recently I read a statement that owing to vibration the *nature* of an iron shaft had changed from fibrous to crystalline! While on this point it is necessary to warn those who are beginning the study of the metallurgy of steel against accepting many of the statements in all but the most recent text-books as to the cause of the effects produced by the different elements and by heat treatment on iron and steel. In point of fact many theories previously considered as well proved have been swept away by the microscope, and at the present time the new and correct ones are not properly established. Many of the leading metallurgists in different countries are, however, working on the subject, and I think that in a few years at most our knowledge of the more common conditions will be fairly complete.

Before taking any example of defective material it will be well to describe the ideal structure of iron and steel. Slide No. 4a shows that of pure iron or ferrite, as it is called, which consists simply of crystals of the cubic system, i.e., each crystal is made up of little square bricks, which all face in the same direction in each individual crystal, but are differently pointed, or as it is technically called, orientated, in different crystals. This is very clearly shown in the next slides (Nos. 5a and 6a). These are from photographs by Mr. Stead, of Middlesbrough, and are of iron containing about 4 per cent of silicon. The structure is, however, precisely the same as that of pure iron, but the silicon enables the crystallization to be more clearly shown. The little crystals seen must not be assumed to be the ultimate structure, as each one consists of smaller ones, and I think it quite probable that if it were possible to go on investigating we might find that the ultimate molecules were also cubic in form, which result would, I believe, help to support Mr. Macfarlane Gray's theory of heat.

In the recent excellent lectures given here by Mr. Milton he pointed out that all metals are crystalline, but that the crystals were generally imperfect as to their boundaries, and that for this reason some people call them grains. It is undoubtedly true that the outlines of most of those found in ordinary metal are not in accordance with that of true crystals, but I think we should not judge of them any more than people by their outward appearance. Inwardly they are just as purely crystal as a diamond; for this reason I myself prefer to speak of them as crystals.

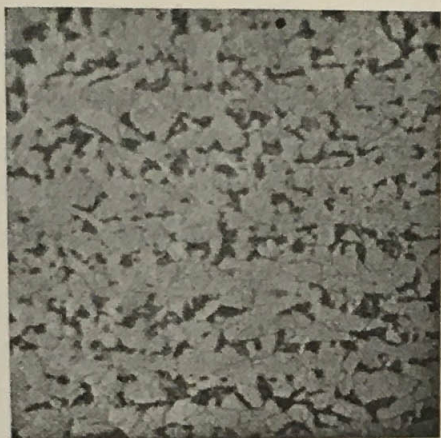
We will now pass on to steel, and the first thing that strikes us in considering this marvellous alloy is why small additions of carbon make such great differences in its strength and qualities. This remained a mystery until its structure was examined under the microscope, up to which time it was, I believe, a popular theory that it consisted of a very uniform molecular mixture of iron and carbon, the latter being possibly in combination with some of the former. Sir Frederick Abel, the eminent chemist, had certainly isolated a carbide of iron, but I believe only from pig iron. Anyhow this carbide has, as it has turned out, proved to be a very important and in fact the only one we know of as a certainty. Its formula is  $\text{Fe}_3\text{C}$ , that is to say, three atoms of iron to one of carbon, and it occurs in all



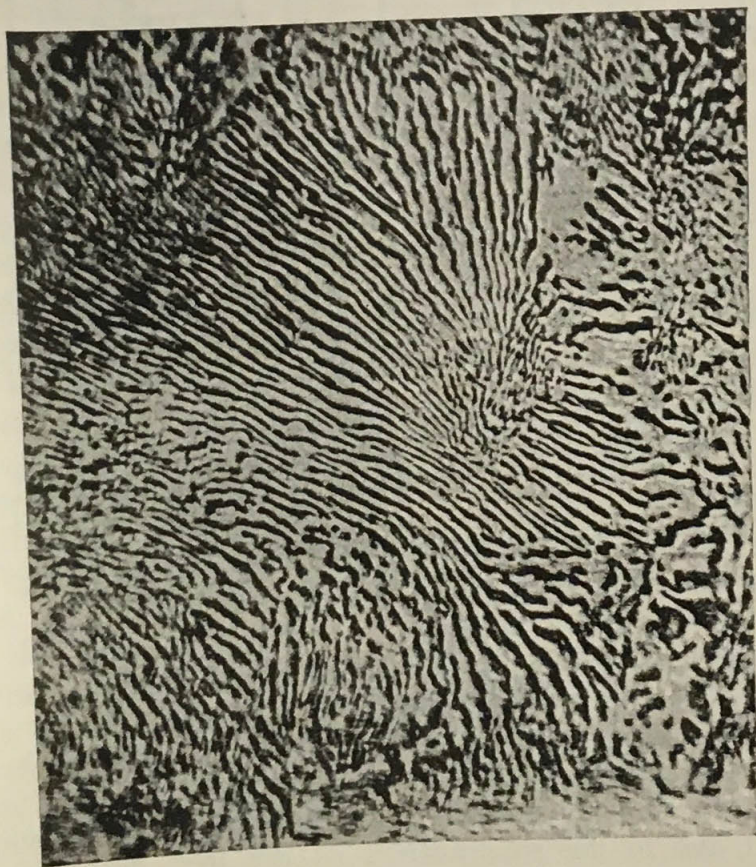
steel in ordinary use. It is extremely hard when in mass — harder in fact than hardened steel — and is now known in metallurgy by the name of cementite. You will notice that the carbon has, as it were, made itself more powerful and larger by uniting with three parts of iron, but this would not be enough to give the actual strength obtained in steel, and consequently we find it in mild steel in small patches of a curious mixture of cementite and iron, which is called pearlite. They are the dark areas of the photograph now shown (No. 1), and you will see that by getting between the iron crystals they stiffen the whole mass up and thus produce the great increase of strength of the steel. I should perhaps mention that the pearlite is not naturally dark, and is only made so by the action of the agent used for etching, the object aimed at being to differentiate it from the iron crystals.

The section from which this photograph was made was taken from a boiler-shell plate which failed in the working, but the defective part is not shown in this slide. In really good material, however, the pearlite would be quite evenly distributed and the size of the crystals would be smaller. This plate contained about .18 per cent of carbon, and the tensile strength was about 28 tons. In proportion as the percentage of carbon is increased so does the area of pearlite until, as first shown by Arnold, when the carbon reaches .89 per cent in pure carbon steel or about .8 per cent in ordinary commercial steel, the whole of the material is composed of pearlite.

The next slide (No. 7a) is taken from steel of this description, which may be called true steel, and shows the pearlite greatly magnified so that its structure may be distinguished. It consists of plates of hard cementite (carbide of iron) alternated with plates of iron; and when viewed in the microscope in a good light this produces an appearance like mother-of-pearl, owing to defraction of light by the thin cementite plates. The name pearlite was in fact given owing to this effect, which was first discovered by Dr. Sorby. The structure now shown is, however, seldom found in practice in mild steel with the exception of well-annealed castings, as when a fair amount of manganese is present the pearlite becomes more or less granular, the hard parts then being, I understand, a double carbide of iron and manganese corresponding to the formula  $(\text{FeMn})_3\text{C}$ . This explanation will, I hope, be sufficient to show how it is



Slide No. 1. — Mild Steel, 58 diam.,  $\text{HNO}_3$ .

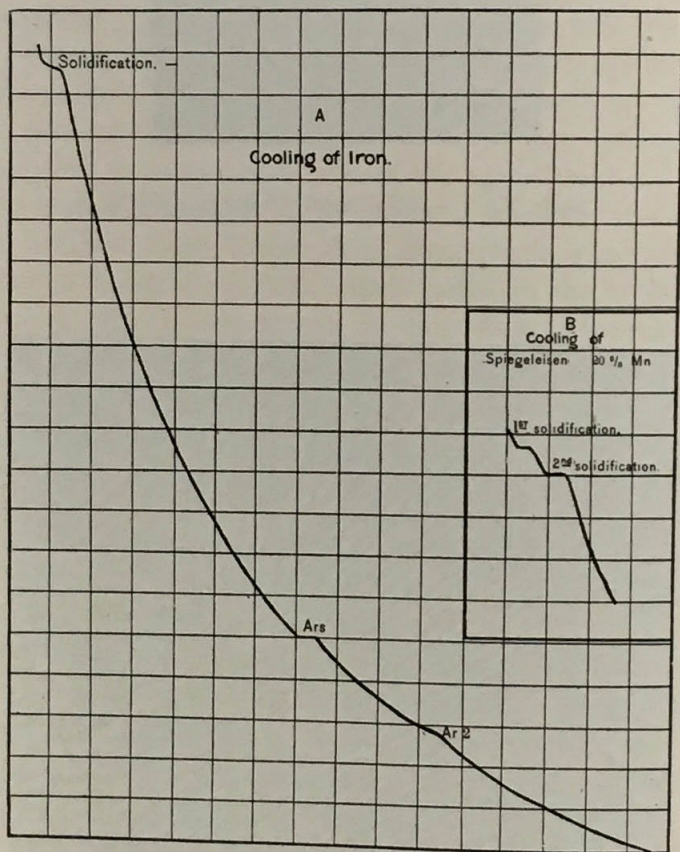


Slide No. 7a. — Pearlite, 1000 diam.



that a small quantity of carbon produces such a marked effect on iron.

There is another important modern method of investigation, which is of considerable help to the microscope, especially when considering the phenomena arising from the tempering or hardening of steel, and that is the use of cooling curves. In the recent lectures by Mr. Milton, that gentleman explained the principle



Slide No. 8a. — Cooling Curve of Pure Iron.

of these curves, and gave some examples of those belonging to certain alloys. It will therefore only be necessary for me to mention that in the diagram No. 8a, which represents the cooling curve of pure iron, horizontal distances represent time and the vertical ones temperatures. At the extreme top a stop has occurred due to the solidification of the iron itself, and you will notice that afterwards, instead of the curve being con-

tinuous, there are two distinct breaks in it, and in the case of mild steel there would be a third. The question naturally arises, what is the cause for these breaks or arrest points, as they are technically called? and I regret to say that, with the exception of the lowest (not shown on this diagram) I cannot tell you. The lowest point is acknowledged to be that due to the formation of the cementite, and at the second iron becomes susceptible to magnetism, but in other respects the meaning of these stops has been and is the subject of fierce controversy between two opposite schools of thought — one called the “allotropists” and the other the “carbonists.” The first follow the lead of M. Floris Osmond, a French engineer, and includes Sir W. C. Roberts-Austen, chemist to the Mint, and other scientific men. The leader of the carbonists is Professor Arnold, of Sheffield, who is supported by Mr. Hadfield, of the same town, and many others. A brief enunciation of the two theories may be of interest. The allotropists assert that the two upper arrest points in this diagram are due to iron changing from one allotropic state to another. Above the top one called  $Ar_3$  they call the iron *Gamma* iron, between  $Ar_3$  and  $Ar_2$  *Beta* iron, and below that *Alpha* iron. Moreover, they assert that the hardening of steel is due to the retention in its allotropic state of *Gamma* iron, the only effect of the carbon being to assist in doing so. If this is so, it may be asked why it is not possible to harden absolutely pure iron, to which they reply that the changes then take place so rapidly that one cannot stop them in time. On the other hand, Professor Arnold and the carbonists hold that the hardening of steel is due to the presence of the carbon, and I will briefly describe his theory later on whilst some mild tempered steel is being shown.

The next curve (Slide No. 9a)\* is due to Sir W. Roberts-Austen, who has so modified the Le Chatelier pyrometer as to make the arrest points much clearer and more evident. The curve in fact really represents the difference between the rate of cooling of a piece of very pure iron and a cylinder of platinum. The cause of the top stop in this case was unknown at the time, but has since been found to be due to the wires used in the pyrometer. The two lower arrest points were discovered by Sir

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\* See *The Metallographist*, Vol. II, p. 194, Fig. 6.



W. Roberts-Austen and are due to hydrogen occluded in the metal. This may appear to be of only purely scientific interest to many of you, but it has a practical bearing. Many years ago Mr. William H. Johnson found that steel wires if allowed to remain in dilute sulphuric acid became brittle, and he ascertained that the cause of this was the occlusion of nascent hydrogen gas in the metal. This fact is of some interest and importance in dealing with galvanized steel, which is usually pickled in sulphuric acid; fortunately, however, it is found that by heating the metal to between  $100^{\circ}$  and  $200^{\circ}$  C. the surplus gas is driven out and the material regains its normal quality. These pieces of steel wire, for which I am indebted to Messrs. Yarrow & Co., exemplify this peculiarity. The first is in its normal condition and can be readily bent; the second has been placed in a solution of 1 part of acid to 10 of water for half an hour and is quite brittle; and the third has been heated to about  $100^{\circ}$  C. in a bath and has regained its ductility. Professor Arnold has also pointed out that the hydrogen arrest points occur at what is commonly called black heat when mild steel is known to lose a certain amount of ductility.

This completes what I may call the first and introductory part of my lecture; the second part, being to a great extent devoted to defects in material, as revealed by the microscope, will I hope be of more interest. I have arranged the different samples approximately according to the percentage of carbon contained in them, and accordingly begin with wrought iron.

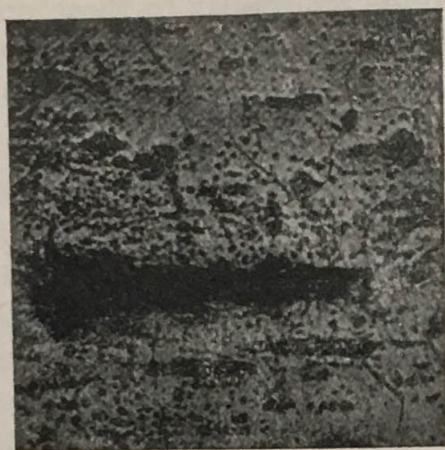
The slide (No. 2) now shown is taken from the lower end of a wrought iron combustion chamber girder stay which broke after being overheated; in fact the chamber top came down, the cause being excessive accumulation of scale. From a cursory glance at the structure, one might be led to think that the material was defective on account of the number of flaws shown. This is not the case, and I here desire to emphatically controvert an impression that I have heard expressed to the effect that metallographists frequently regard material as defective owing to small flaws appearing big when highly magnified. Ordinary slag or sulphur flaws are as a rule negligible as far as longitudinal strength is concerned, but of course if the material is stressed at right angles to their direction a considerable diminution of strength and especially of elongation occurs. All puddled



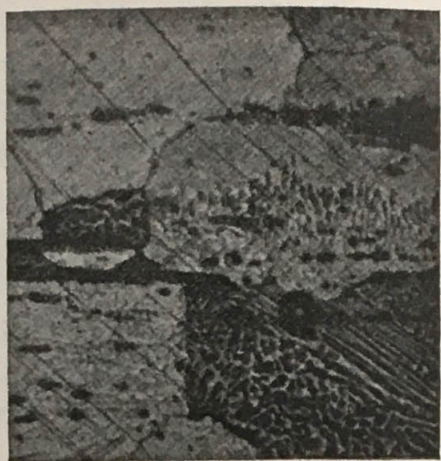
iron contains oxide of iron and slag flaws similar to those now shown, and to this cause the strength is always less across the grain as it is commonly called. You will observe, however, that this is a somewhat misleading expression, as the crystals of iron are fairly symmetrical unless the material is rolled at a very low heat, and that the so-called fibres are only due to the flaws. These also frequently cause the iron to break with a fibrous fracture, but you must not on that account assume that they are a good feature. The fact is that, as previously stated, nearly all iron good or bad contains more or less of them, but in good material the crystals of iron themselves are so ductile and the coherence between them is so good that they stretch and elongate until the flaws come so close together that fracture occurs. With bad material on the other hand the crystals are brittle and large; they do not stretch to any extent and fracture occurs through them giving a crystalline appearance. Perfectly pure iron is an extremely difficult substance to get owing to the high temperature at which it melts, at which temperature it is practically impossible to prevent it absorbing impurities. Some that has been made, however, at the University College, Sheffield, proved to be as soft and ductile as copper, and when broken in the testing machine it drew down to a pencil point. I hope these remarks are sufficient to make the following points clear to you — that all iron is crystalline; that absolutely pure iron is similar in ductility to copper; that commercial wrought iron contains a large number of oxide of iron and slag flaws, which when the iron itself is fairly pure cause a fibrous fracture; when impure (although the flaws are still there) the fracture passes through the crystals and the metal appears crystalline.

I will now show you an example of a very impure iron (No. 3). This is from a stay tube of a new boiler, which broke under the hydraulic test. The crystals are abnormally large and quite visible to the naked eye after being etched, many of them being quite  $\frac{3}{16}$  in. long, and this is the metal whose structure I mentioned as being easily seen without the microscope, in the early part of the lecture. The cause of these abnormal crystals is chiefly phosphorus, of which the iron contains .3 per cent, but the metal has also been worked at an improper temperature. The tubes were exceedingly brittle, and the fracture was of a coarse crystalline character. Mr. Stead has shown, as was

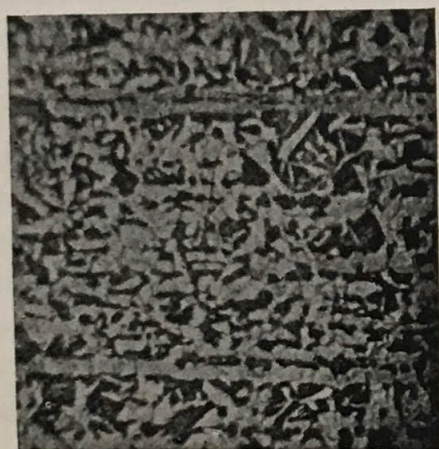




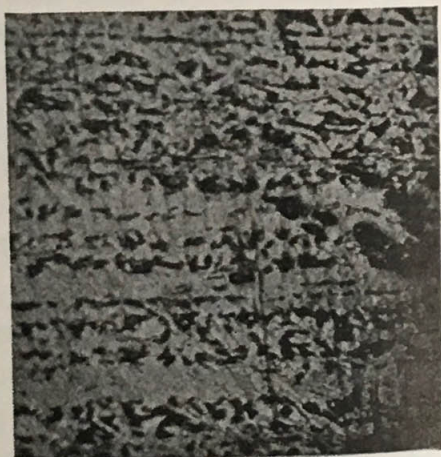
Slide No. 2. — Wrought Iron Stay,  
53 diam.,  $\text{HNO}_3$ .



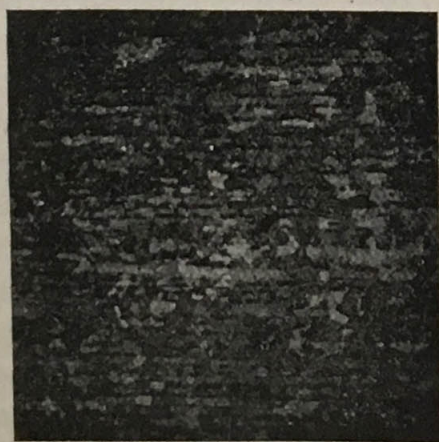
Slide No. 3. — Phosphoretic Iron  
Stay Tube, 58 diam.,  $\text{HNO}_3$ .



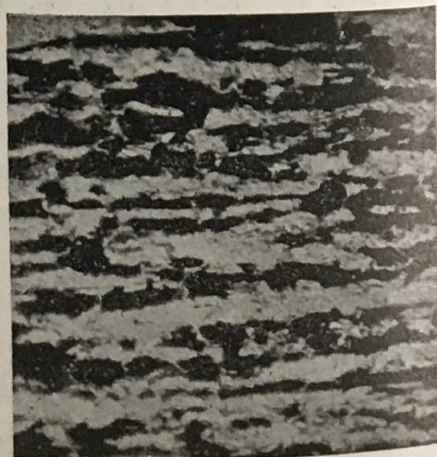
Slide No. 4. — Mild Steel Boiler Plate,  
near outside, 58 diam.,  $\text{HNO}_3$ .



Slide No. 5. — Mild Steel Boiler Plate,  
near centre, 85 diam.,  $\text{HNO}_3$ .



Slide No. 6. — Mild Steel Boiler Plate,  
 $\text{HNO}_3$ .



Slide No. 7. — Over-Annealed Steel  
Bar, 53 diam.,  $\text{NH}_4\text{NO}_3$ .



described by Mr. Milton in his lecture, that phosphorus below 1.7 per cent was in solution in the crystals of iron (when carbon was absent), but from the markings seen in one of the crystals it would seem as if there were some sort of a eutectic present in this metal which contains small quantities of carbon, silicon and manganese. I have also noticed somewhat similar peculiar markings, which require further investigation, in several specimens of iron containing an unusual amount of phosphorus. I would now specially draw your attention to these lines, which are not scratches but are called slipbands, and are produced by the straining of the metal when cold; in this case by the rolling of the tube in the tube-plate. You will remember that I showed you that each crystal of iron was made up of a number of small ones symmetrically arranged. Now when a metal is strained what occurs is this. The different rows of tiny crystals begin to slide over each other, and this is how what is called the flow of metals takes place. When such metal is polished and etched the acid eats in between some of the rows and produces the lines you see, and you will notice that these lines are always parallel in each crystal but are at different angles in different crystals. Professor Ewing, of Cambridge, was, I believe, the first to discover these facts, and has given many beautiful examples of strained metals. I should, however, mention that as a rule iron and mild steel, possibly owing to their ductility, are not favorable subjects for this study.

We will now take some examples of mild steel, the most important material with which we have to do. This photograph (No. 4) is taken near the surface of a mild steel boiler plate which cracked whilst being bent in the rolls. It was over  $1\frac{3}{8}$  in. thick, and the test results gave about 30 tons, with over 20 per cent elongation in 10 in. The dark part of the photograph is the end of a crack, which did not penetrate further, and at the top may be seen the effect produced by hammering the surface of the plate. Owing to the action of the acid and the comparatively low magnification, it is not possible to see any slipbands in this part. With regard to the cause of failure in this instance I hope to give you an explanation when taking a subsequent case, but before passing on I want you to particularly notice that the material is not very homogeneous, and also to note these bands of carbonless iron. With respect to the evil effects of cold ham-



mering, it does not require any special knowledge to see that the crystals are so crushed and bruised that very little adhesion can be left; consequently, if the plate is bent so that these parts are on the convex side a crack is started, and this may easily spread through the whole plate. I also want you to get a rough idea of the proportion of pearlite, i.e., of the carbon to the iron, in order to compare it with that shown in this next view (No. 5), which is from near the centre of the same plate, where there is perhaps four or five times as much carbon. Now this is a very important point, as it shows us that this plate, instead of being homogeneous as one might expect, is composed of steel which may only have a strength of 24 tons per square inch on the surface, and perhaps 36 in the centre. I do not say that from a practical point of view it is any the worse for this peculiarity — indeed, it may be more ductile — but it is just as well to know the facts, and I think it would be interesting to have a series of experiments carried out with metal of this description in order to definitely ascertain its qualities as compared with homogeneous material. The reason for this variation is doubtless segregation of the carbon towards the centre of the ingot from which the plate was rolled, and it is an example of the danger of relying on a chemical analysis without examination of the structure of the material. The metal is somewhat lamellar or streaky — a point of considerable importance, and respecting which a very marked example is given in the next slide.

Slide No. 6 is also from a boiler-shell plate, the strength of which was a little over 28 tons and the elongation good. The structure very closely resembles that of a brick wall, and it is without doubt a very undesirable one, and one which is specially liable to produce failure under vibration. It was in 1899 that I had this metal under examination, and in 1900 a paper was read at the Institution of Naval Architects by Signor Schanzer, of the Terni Steel Works, Italy, describing a similar structure in a large steel shaft which fractured. He attributed, and I think rightly, the failure of the shaft to this reason, but did not express an opinion as to the cause which produced the structure, though he mentions that possibly phosphorus, which was somewhat high in this case, may have had something to do with it. (In the boiler plate now shown the phosphorus was about .06 per cent.) I am myself inclined to think that the cause will be found in the



structure of the ingot, which may be similar to that of a coarse honeycomb, the cell walls being chiefly of pearlite, and the cells themselves filled with iron crystals. I will show you later a photograph of such a structure. When therefore the metal is rolled the iron crystals are flattened out into the layers you now see. Another explanation of this form of structure, which applies in certain cases, has been given by Professor Arnold, and is as follows. If a piece of rolled metal be strongly annealed or very slowly cooled, as, for instance, in the same manner as a steel casting, the pearlite and ferrite will arrange themselves in layers parallel to the direction of rolling, and this will be the case even if previously no such a structure existed. Professor Arnold attributes this to a crystalline habit impressed on the metal by the work done on it. I shall now show you an example of this. This slide (No. 7) represents the structure of a medium steel bar 1 in. square, which a steelmaker had kindly annealed with some mild steel castings; the duration of annealing would therefore be about three days. The structure is of a very lamellar character, and although the tensile tests gave good results, yet such material would be very unreliable under vibratory stresses. In the absence of any approved mechanical tests for finding out the inferiority of this material an examination of the structure under the microscope appears to be the only method at the present time for detecting it.

The next slide (No. 8) is also from a mild steel plate which failed; this is from near the surface. The most noticeable features are the broad carbonless bands, which are also spotted with sulphur and other flaws, and which are, in my opinion, nearly as deadly in their effects on steel as the "spotted band" described in one of the adventures of Sherlock Holmes was to human beings.

I will now deal briefly with some of the causes which result in the so-called mysterious failures of steel plates. In the first place it may be noted that such plates are frequently those of large size and great thickness; this results in comparatively little work being done on them, and the crystals of iron are independent of each other instead of interlocking as they do in steel which has been well worked. The same cause also results in the plate leaving the rolls at a temperature much above the normal, causing the iron crystals to be of excessive size. But frequently the most



important cause is, in my opinion, the following, which I have long suspected, but confirmatory proofs of which I have only lately obtained; as far as I know it is quite a new theory. I attribute many of these failures to the presence of carbonless bands as shown in this photograph, which bands, I believe, contain a relatively large proportion of phosphorus, and are very much inferior in ductility to the remainder of the metal. They are generally near the surface, and when one actually outcrops, what is called a surface defect is frequently produced. Now when a plate is bent in the rolls and one of these outcrops is reached the inferior metal cracks, and a crack once started will, as you know, go to a considerable depth. At the same time if such a plate is broken in the testing machine the ductility of the remaining metal conceals that of these veins of bad metal; similarly, although the analysis generally shows that the phosphorus is fairly high, yet it does not indicate that a good deal of it is concentrated in one part. I do not, of course, pretend that all failures of plates can be attributed to this cause, but it will, I think, be sufficient to explain a good many cases which have seemed mysterious.

The next slide (No. 9) shows the metal at the centre of the same plate; there is a large increase in the amount of carbon and the structure is rather lamellar. I will now show you some examples of the effect of heat treatment on steel, which of course produces a considerable difference in its properties, but of which a chemical analysis will give hardly any information.

This slide (No. 10) is taken from another boiler plate which failed, the causes being carbonless bands (not shown in this photograph) and finishing at too high a temperature, which has resulted in the iron crystals being abnormally large. In the next slide (No. 11) we have a piece of the same material which has been raised to a full red-heat (above the upper arrest point) and allowed to cool quickly in the air. You will notice the enormous difference in the size of the crystals of the body, but in the carbonless bands the size is not much altered owing to the phosphorus present; it would therefore appear that a more drastic annealing was necessary in order to remove the effects of the deleterious parts. With regard to the size of the crystals shown I do not wish it to be understood that those of a boiler plate could be made so small in practice, because it would not be possible



or desirable to cool it quickly enough, but there is little doubt that those seen in the preceding photograph could have been reduced in size at least one half and the material thereby increased in toughness. You will notice by comparing this and the photograph of the steel bar that whilst prolonged annealing is injurious to forged steel, raising it to a temperature above the highest arrest point and allowing to cool in air is, as a rule, of great value in making it fine grained and thereby improving the qualities of the material.

The next example (No. 12) is a piece of the same plate which has been heated above the lowest arrest point and quenched in water. This naturally brings us to the question of temper bends, in which the steel is generally required to be heated to a cherry red and quenched in water. Well, cherry red is a somewhat vague expression, but I may point out that unless the temperature to which the steel is raised and quenched is above that of the lowest arrest point, or really the corresponding acceleration point, which is not quite identical and is about  $680^{\circ}$  C. in mild steel, practically no change takes place, and the test is no more trying than if performed with the steel in the normal condition, with which, indeed, it is nearly identical. Now, it is somewhat difficult to measure temperatures exactly with the eye, but the temperature just mentioned corresponds with what careful observers have called a full cherry red. To produce any appreciable effect, therefore, on the steel it is necessary to make sure that this temperature is exceeded. I do not think that this is always the case; in fact, some time ago I received, through the courtesy of one of the leading steelmakers, some pieces of what he called typical boiler plate, which included a temper bend made at his works. The material was undoubtedly very good, but there was absolutely no difference between the tempered material and that in the normal condition.

To return to the present sample, you will notice the pearlite has somewhat extended and assumed a ropy appearance. This is called troostite by the allotropists, but according to Professor Arnold it is a form of martensite.\* Now this is usually con-

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\* Professor Arnold has since stated that he calls this constituent "hardenite"; this is, however, only an alteration of the name, the theory remaining the same. With regard to what is usually called martensite, he states that this a structure and not a constituent.



sidered to be the constituent of hardened steel, and as we do not know its composition at present I do not propose to say much on the different theories regarding it. I desire, however, to let you know what is Professor Arnold's theory respecting the hardening of steel, and which is, to a certain extent, illustrated by this and the following slides. This theory is, I believe, that at the lowest arrest point the compound constituent pearlite changes into martensite, and that as the temperature increases this spreads out and finally permeates the whole mass. This is certainly a simple theory, and it is to be hoped, if only for that reason, that it is correct. The whole matter will, however, be probably thrashed out at the forthcoming meeting of the Iron and Steel Institute.

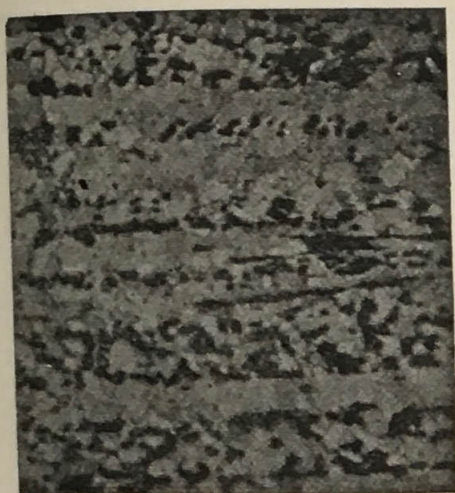
In the slide now shown (No. 12) it is evident that the dark etching constituent is extending, and in the next, which is the same steel quenched from a still higher temperature, it has diffused still more.

This slide (No. 14), which is at the same magnification as the previous ones, represents the metal at one end of a comparatively new thrust shaft which failed. You will notice the very coarse structure of the metal, as evidenced by the size of the pearlite areas, but owing to the method of preparation in this case the iron crystals cannot be clearly seen. Nevertheless the cause of failure was not due to this structure, and, in fact, a test made from this end of the shaft gave 31 tons' tensile strength with good elongation.

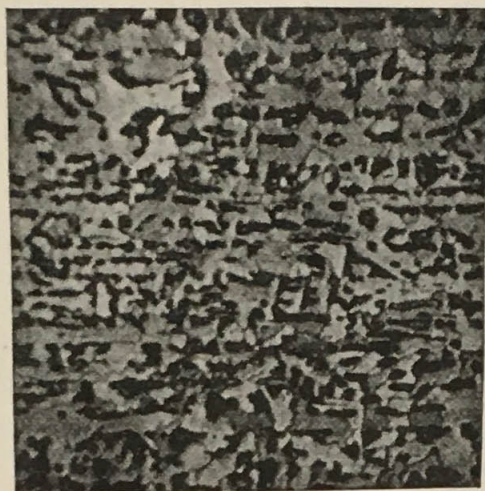
The next photograph (No. 15) is taken near the point of fracture of the same shaft, and has a completely different appearance. The general structure is so coarse that it is only partly represented in the photograph, and it really consists of large cells of iron crystals surrounded by meshes of mild steel, and is the same in any plane of section. I may mention that the crossed structure shown is a characteristic of unannealed cast steel as distinguished from annealed metal. From an examination of this steel I have no hesitation in saying that this shaft was a casting and was not forged at all; in fact, only one end of it was annealed, and the cause of its fracture must be ascribed to these features.

The next slide (No. 16) represents a brittle mild steel casting, and is taken from the end of a tensile test piece. The struc-

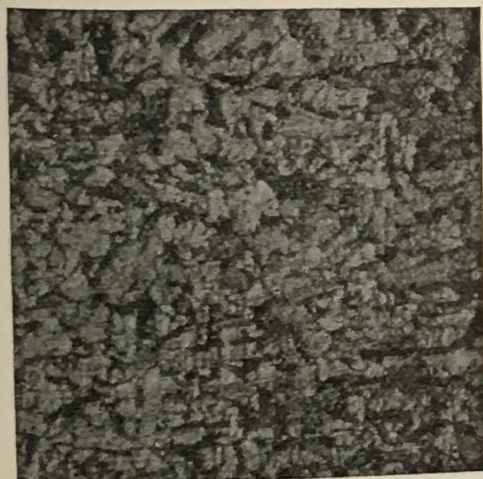




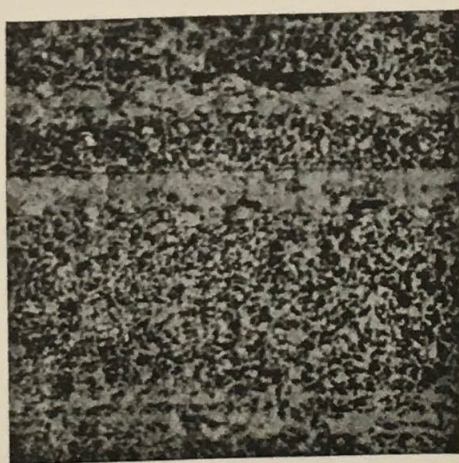
Slide No. 8. — Mild Steel Boiler Plate, near outside, 58 diam.,  $\text{HNO}_3$ .



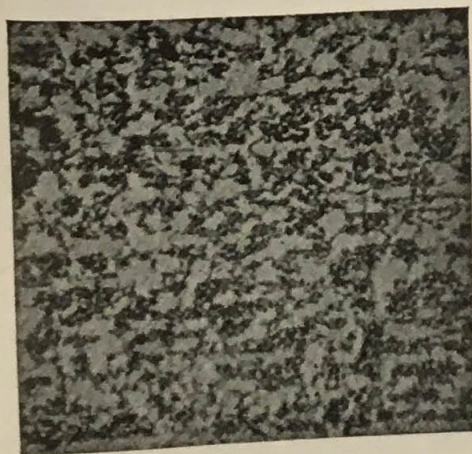
Slide No. 9. — Mild Steel Boiler Plate, near centre, 58 diam.,  $\text{HNO}_3$ .



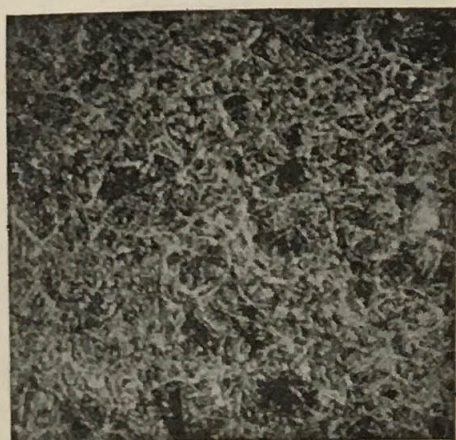
Slide No. 10. — Mild Steel Boiler Plate, normal, 53 diam.,  $\text{NH}_4\text{NO}_3$ .



Slide No. 11. — Mild Steel Boiler Plate, annealed, 53 diam.,  $\text{NH}_4\text{NO}_3$ .

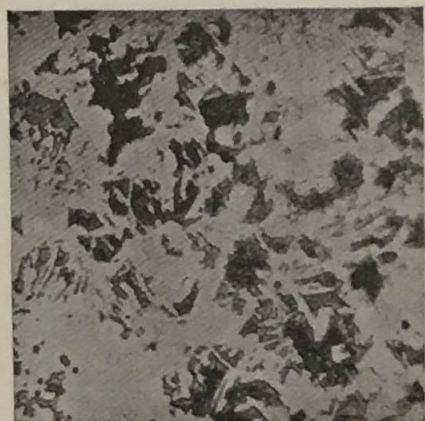


Slide No. 12. — Mild Steel Boiler Plate, tempered, 53 diam.,  $\text{NH}_4\text{NO}_3$ .

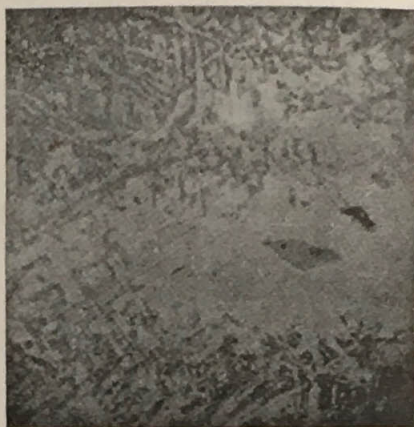


Slide No. 15. — Mild Steel Boiler Plate, tempered, 53 diam.,  $\text{NH}_4\text{NO}_3$ .

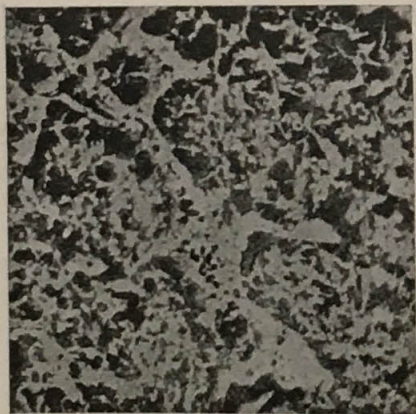




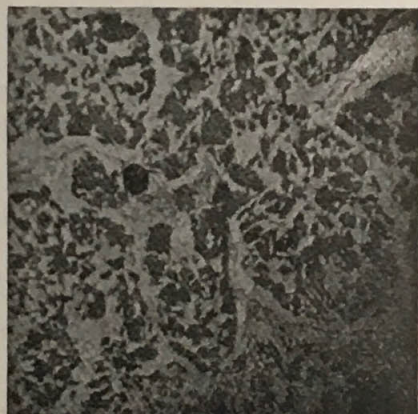
Slide No. 14. — Mild Steel Thrust Shaft  
Flange, 53 diam.,  $\text{NH}_4\text{NO}_3$ .



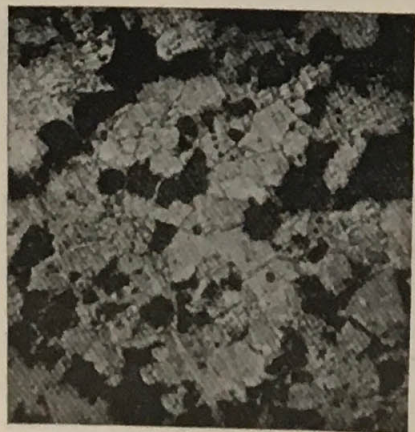
Slide No. 15. — Mild Steel Thrust Shaft,  
at fracture, 58 diam.,  $\text{NH}_4\text{NO}_3$ .



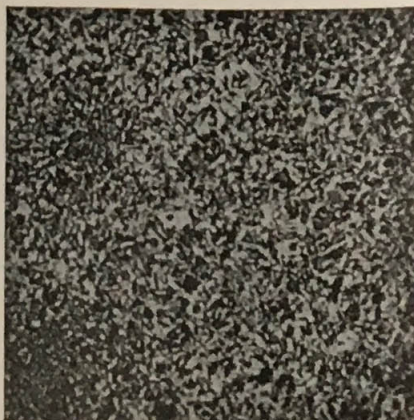
Slide No. 16. — Brittle Mild Steel  
Casting, 53 diam.,  $\text{NH}_4\text{NO}_3$ .



Slide No. 17. — Brittle Mild Steel  
Casting, 58 diam.,  $\text{NH}_4\text{NO}_3$ .



Slide No. 18. — Well Annealed Mild  
Steel Casting, 58 diam.,  $\text{HNO}_3$ .



Slide No. 19. — Nickel Steel,  
53 diam.,  $\text{NH}_4\text{NO}_3$ .



ture shown is not unusual in imperfectly annealed castings, and, generally speaking, consists of cell walls of iron, which enclose a mixture of pearlite and iron crystals. The cause of brittleness in this case is due to the flaws of sulphide of manganese, which you see in the middle of the cell walls. This was first pointed out by Professor Arnold, and although the flaws in this case are not continuous they probably were before annealing. It should, however, be pointed out that defects of this nature generally only occur in small castings, the big ones, such as the shaft just considered, generally containing sufficient heat to overcome this source of weakness.

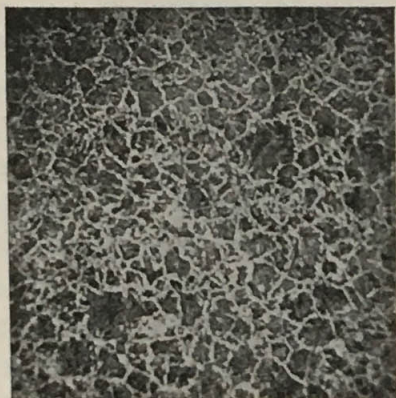
The photograph now shown (No. 17) is from the same piece of steel but is taken only about  $\frac{1}{8}$  in. from the fractured end. You will see that the metal has begun to part and that the fracture passes along the cell walls, showing the weakening effect of the flaws previously mentioned. This steel gave only 9 per cent elongation.

From some different samples of mild cast steel that I have examined it would seem that insufficient annealing is far from being infrequent, and the only perfect structure I have is, I regret to say, from steel made by a foreign firm. However, the structure shown in the next slide (No. 18) is from steel by a British maker, and is very good for cast metal. I do not wish to infer that British makers cannot make as good or better steel than anyone else; only, in some cases, sufficient care has not been taken with respect to the annealing. I am now going to show you photographs of two of the very best medium carbon steels that, I think I may say, the world can produce. They are by two Sheffield firms, and the steel was ordered by a celebrated firm of torpedo boat builders, not unknown in this neighborhood. I understand the order was for the very best nickel steel of about forty tons' strength regardless of price, which, I am told, was a very stiff one indeed.

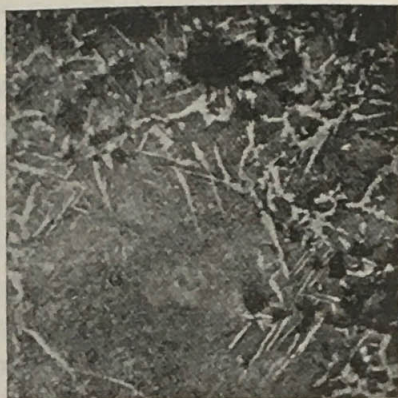
Slide No. 19 represents the nickel steel supplied by one firm, and if one only regarded the proportion of pearlite the metal would not appear to possess such great strength. The action of nickel on the structure of steel has not been very fully investigated, but, as far as I know, it reduces the size of the crystals and makes them pointed and interlocking.

The next photograph (No. 20) represents the steel supplied

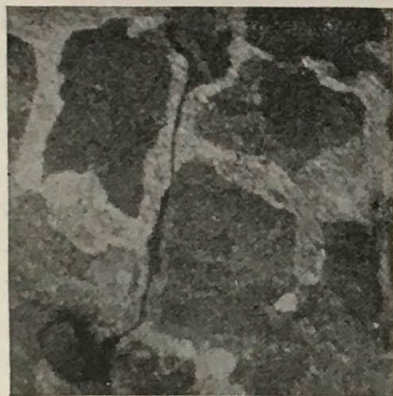




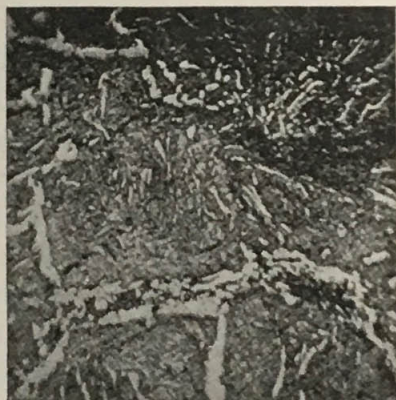
Slide No. 20. — Medium Steel,  
 $\text{NH}_4\text{NO}_3$ .



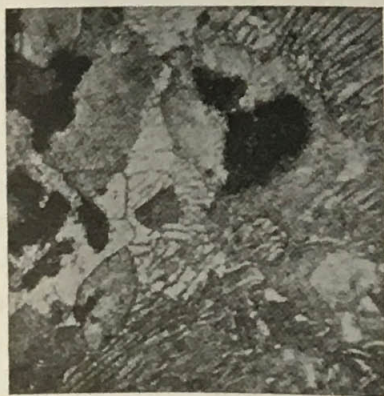
Slide No. 21. — Metal found inside a  
Forging, 24 diam.,  $\text{NH}_4\text{NO}_3$ .



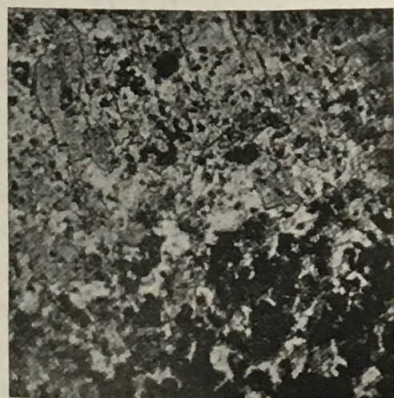
Slide No. 22. — Metal found inside a  
Forging, 236 diam.,  $\text{NH}_4\text{NO}_3$ .



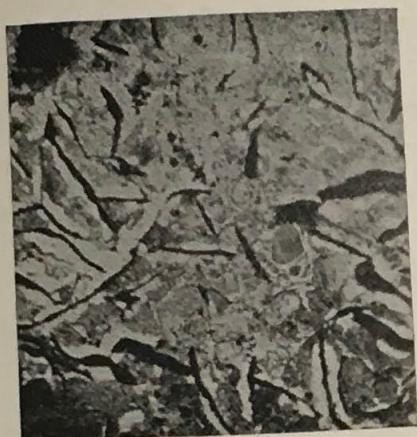
Slide No. 23. — Blister Steel,  
58 diam.,  $\text{HNO}_3$ .



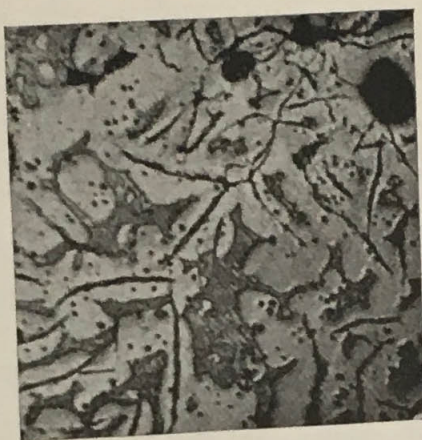
Slide No. 24. — High Carbon Steel,  
236 diam.,  $\text{HNO}_3$ .



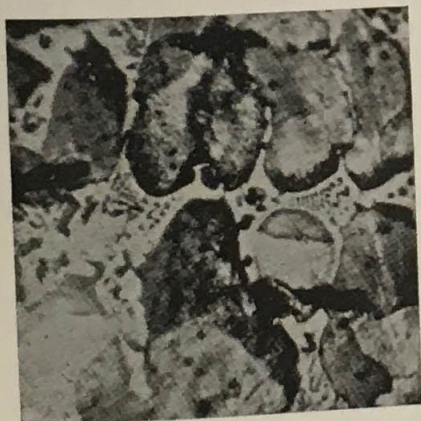
Slide No. 25. — Malleable Cast Iron,  
236 diam.,  $\text{HNO}_3$ .



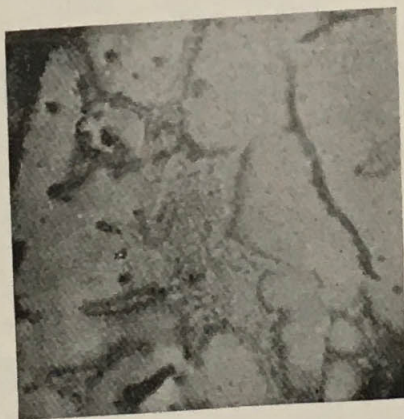
Slide No. 26.— Cast Iron,  
58 diam.,  $\text{HNO}_3$ .



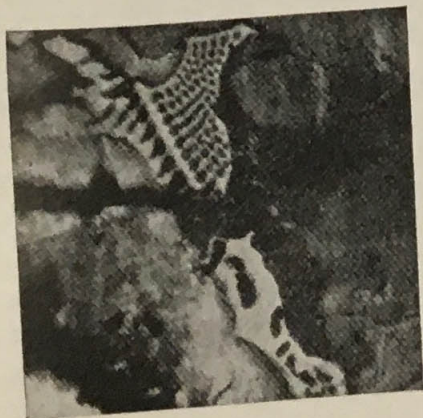
Slide No. 27.— Phosphorectic Cast Iron,  
75 diam. Heat tinted.



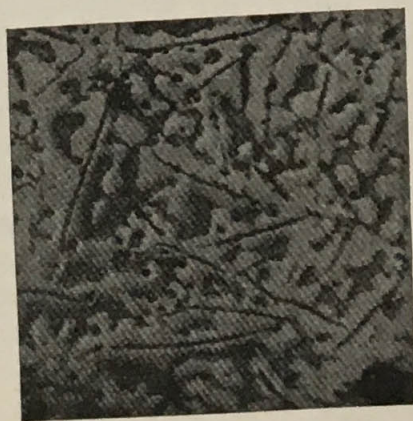
Slide No. 28.— Phosphorectic Cast  
Iron, 236 diam.,  $\text{HNO}_3$ .



Slide No. 29.— Phosphorus Eutectic,  
188 diam. Heat tinted.



Slide No. 30.— Phosphorus Eutectic,  
385 diam.,  $\text{HNO}_3$ .



Slide No. 1b.— Cleveland Pig Iron,  
58 diam. Heat tinted.



would be quite rotten, but being at different angles any pulling stress placed on the metal has a certain amount of solid iron to break. Graphite can, however, resist a very great compressive stress, and it is to this cause that cast iron is so strong to resist compression as compared with tension. Round the graphite plates we find crystals of iron, and beyond them is pearlite, whilst after that we have what is called a phosphorus eutectic. I will describe the nature of this substance when dealing with the next metal, but before doing so I would call your attention to the manner in which this structure illustrates what happens when molten cast iron cools. In the first place the graphite separates out, then come the iron crystals, which group themselves round the graphite plates, and after that the combined carbon, at first in the form of martensite, which afterwards changes to the pearlite shown. Finally at about  $900^{\circ}$  C., the phosphorus eutectic solidifies, but this has by that time been forced by the other constituents to take up the vacant spaces left, and we therefore find it, as a rule, furthest from the graphite plates.

In the metal now shown (Slide No. 27), also cast iron, there are only three constituents — graphite, iron and phosphorus eutectic. The absence of pearlite is due to the fact that there is a large amount of silicon present which has the power of keeping the carbon in the graphitic state, and the metal is, of course, much softer to file or turn. The silicon appears to be dissolved in the iron, and does not, unless in great quantities, make any marked difference in the structure. In the slides, however, shown at the beginning of the lecture, where there was 4 per cent silicon, the crystals of iron seemed to resist the action of the acid better, and there is no doubt that a fairly large proportion of silicon makes the iron crystals more brittle. Now with regard to the phosphorus eutectic. In the first place, I should say that the word eutectic is applied to those structural constituents which solidify last; they must also not be in proportions for chemical combination, and generally speaking, they consist of two constituents which may themselves be of compound character. These, as a rule, arrange themselves in parallel lines or dots. Thus pearlite is the eutectic of steel, and, as before mentioned, it consists in its simplest form of thin parallel plates of cementite separated by similar plates of iron. In the iron now shown the phosphorus amounted to 1.4 per cent, and as this eutectic happens



to be of a brittle character the iron was rendered very weak in consequence. I would here address a word of warning against judging cast iron by its fracture. Apart from other characteristics, it is practically impossible to tell by this means whether the phosphorus it contains is in dangerous proportions or not. As a matter of fact, the iron now being dealt with had a most clean, close-grained and sound appearance at the fracture; yet it was most inferior as regards strength on account of the phosphorus and silicon it contained. Now phosphorus has several advantages from a founder's point of view—for instance, it increases the fluidity of the metal and assists to make sound castings. It is, therefore, an element that requires to be carefully watched for and kept within reasonable quantities, say .3 per cent, in castings exposed to steam pressure, and particularly where any shocks due to water-hammer, etc., may occur.

Slide No. 28 shows an enlarged view of some of the same metal; the continuous line of eutectic can be seen, but the most striking feature is this diamond-shaped crystal in the corner. This is, I believe, according to Mr. Stead, who has written the standard work on this subject, a rhombic crystal of phosphide of iron ( $\text{Fe}_3\text{P}$ ). This is very rare in ordinary iron and may be regarded as a curiosity.

Slide No. 29 is a more highly magnified photograph of the phosphorus eutectic. In this case the metal has not been treated with acid. Some graphite plates are to be seen.

Another photograph (No. 30) of the same subject, but treated with nitric acid, has revealed the outlines of the crystals of iron; at the same time it has more clearly marked the eutectic itself. The structure of this, which is of a characteristic nature, is very similar to that of the backbone on an animal, and considering how necessary phosphorus and iron are to our bodies, it has been suggested to me that there may be some connection between the two.

It should be mentioned that the normal composition of the eutectic is, according to Stead, about 10 per cent of phosphorus and 90 per cent iron, and it solidifies at about  $900^\circ \text{C}$ .

Slide No. 1b, for which I am indebted to Mr. Milton, represents some Cleveland pig iron and is of similar structure to that just dealt with, but contains rather more phosphorus, viz., 1.6 per cent. The metal consists of graphite plates and globules,



iron containing silicon, etc., in solution, and the phosphorus eutectic.

Slide No. 31 is a photograph of Carron pig iron, which has the same constituents as the last example, but the phosphorus is only about 1 per cent. The most striking feature is the size of the graphite plates which are plainly visible to the naked eye and which caused the metal to break with a coarse crystalline fracture. This is, however, merely because the iron has been very slowly cooled, and the metal itself is of better quality than the Cleveland iron, which, it may be remarked, broke with a fine gray fracture. It should also be noticed in this case that the graphite plates are very straight, which is another source of weakness. The phosphide areas are small and disconnected and although the proportion is higher than is desirable, still it would not probably greatly alter the tensile strength, though if subjected to shock, the effect might be serious.

Slide No. 32 represents pig iron used in the basic process and contains about  $3\frac{1}{2}$  per cent of phosphorus, whilst all the carbon is in the combined form as cementite, there being no graphite present. The brilliant white parts are plates of cementite, the dark parts pearlite, and the half-tone areas a sort of compound eutectic of pearlite and phosphorus, the nature of which is not exactly known. It will be noticed that there are rows of pearlite running across the photograph at different angles. I am inclined to think that these are the borders of large crystals, and what we see is really a secondary or sub-structure. The metal is extremely hard, and sections can only be prepared with the assistance of an emery wheel.

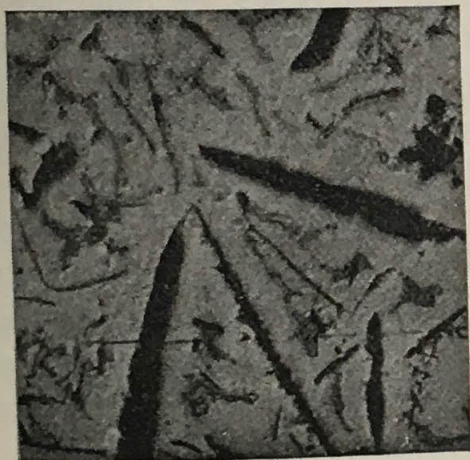
Slide No. 33 is spiegeleisen, which, as you know, is an alloy used for adding carbon and manganese to molten steel. It contains 5 per cent of carbon and 20 per cent of manganese. The broad bands are probably the same double carbide of iron and manganese that forms the cementite in the pearlite of ordinary mild steel. The dotted part is a eutectic of some description.

Slide No. 34 represents the structure of silico spiegel, an alloy used for adding silicon to molten steel; it contains 11 per cent of silicon. The markings seen are eutectic very similar in character to that of phosphorus in the cast irons shown, though there is much more difficulty in etching it.

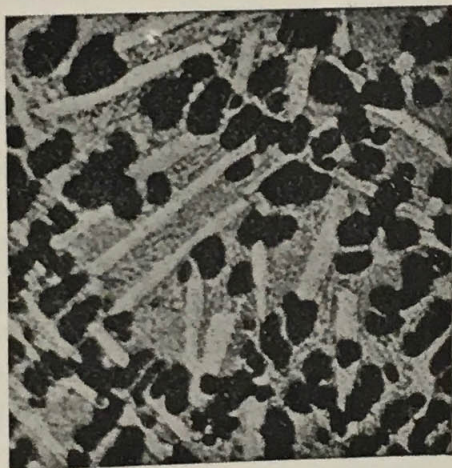
This is the last slide I have to show. I am afraid the par-



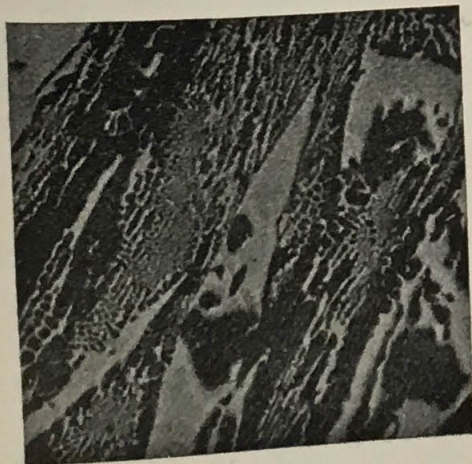
ticulars I have given are of a rather cursory and superficial nature, but I trust they have been sufficient to let you see the advantages of examining the structure of metals instead of relying solely on analysis. Very much remains to be done, and we are still only on the threshold of the temple of knowledge as far as metals are concerned.



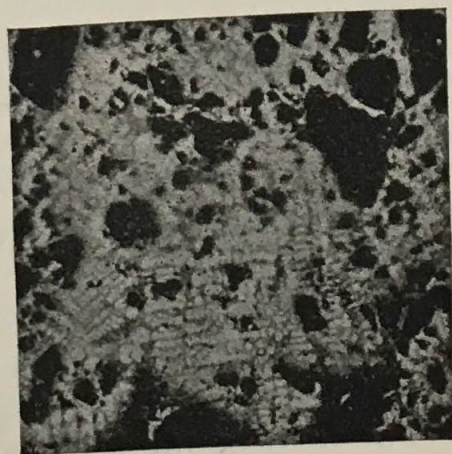
Slide No. 31. — Carron Pig Iron,  
53 diam. Heat tinted.



Slide No. 32. — Basic Pig Iron,  
53 diam.,  $\text{HNO}_3$ .



Slide No. 33. — Silicospiegel,  
53 diam.,  $\text{HNO}_3$ .



Slide No. 34. — Silicospiegel,  
31 diam.,  $\text{HNO}_3$ .

However, as before remarked, there is every probability that the next few years will result in real progress being made, which we shall not have to retract, as has been the case with so many theories propounded before the structure of metals was revealed by the microscope.



## UPON THE STRUCTURE OF METALS AND BINARY ALLOYS\*

By WILLIAM CAMPBELL

**I**N the following paper the author has tried to illustrate the constitution of binary alloys by a few notes upon the structure of metals and alloys. The methods of obtaining a knowledge of the structure of metals and alloys are first discussed, then the crystalline structure of metals, the effects of strain and the effect of heat treatment are illustrated. Finally, the various groups of binary alloys are set forth and the constitution of several examples, representative of the various groups as far as possible, is shown by contrasting or rather by drawing a parallel between their cooling curves and microstructure. The whole of the original work of the paper was carried out at the Royal School of Mines, London, and in the Metallurgical Department of Columbia University.

The magnification and illumination of the various photographs of the structure of metals and alloys are represented by the symbol in the lower right-hand corner, thus: x 33. v. means multiplied thirty-three diameters and vertically illuminated; or x 16. o. means multiplied sixteen diameters and obliquely illuminated.

### PRACTICAL METALLOGRAPHY

Practical metallography may be divided into several distinct parts:

- (1) Preparation of alloys or metal to be examined.
- (2) Preparation of surface.  
Cutting and polishing.
- (3) Etching the prepared surface: development of structure.
- (4) Microscopical examination.  
Mounting the specimen.  
Microscope and accessories.
- (5) Microphotography.

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\* *Journal of the Franklin Institute*, July, August and September, 1902. This article and illustrations are reproduced here through the courtesy of the Franklin Institute.



It would be out of place to do more than glance at one or two of the main points in preparing and examining sections, because the subject has been carefully treated by Stead, Le Chatelier, Sauveur and many others. One of the most important points is the preparation of the alloy or metal, because its final structure will in almost every case depend upon its rate of cooling, the uniformity of cooling, prevention of oxidation, etc., and, in the case of an alloy, upon the thorough mixing of its constituent metals when liquid. As a general rule, the slower the cooling the larger the structure. In many metals and alloys, this holds good not only during solidification, but also afterwards, because profound changes often take place long after solidification. As examples, one immediately thinks of iron and its alloys, of the bronzes and of the brasses. As an example of the necessity for guarding against oxidation, we need only consider copper and the rapidity with which its oxide diffuses through it.

There are several devices to insure slow and uniform cooling. Where a gas furnace is used, automatic cut-offs give good results. Heycock and Neville\* have invented one which will give any desired rate of cooling. It consists of a tube some 15 centimeters long by  $2\frac{1}{2}$  centimeters wide, and contains an inner tube made of many layers of fine silk wound round the outside of a cylinder of coarse iron wire gauze to give it rigidity. With the exception of a line about 2 millimeters wide, running from top to bottom of the silk, the whole of this inner tube is coated with a very viscous shellac. The silk tube is open at its lower end, which dips into mercury, whilst its upper end is cemented to a glass tube connected with the gas supply from a regulator which supplies the gas at a constant pressure. The outer tube has an opening at its base, through which mercury is admitted, and an exit pipe near the top through which the gas passes to the furnace. Thus, the gas from the regulator enters the inner tube at the top, and passes through that part of it which is not covered by shellac into the outer tube and thence to the furnace. If mercury is forced into the outer tube, the gas is slowly cut off. A uniform supply of mercury is obtained from a cylinder above which is a head of water. This head of water is increased

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\* *Trans. Chem. Soc.*, 1898. No. 73, p. 714.



at a uniform rate by means of a constant level water dropper whose supply can be increased or decreased at will. Thus, as the head of water slowly and uniformly increases, the pressure on the surface of the mercury increases, causing it to flow into the cut-off tube and rise inside and out of the silk tube, and hence to gradually cut off the escape of gas through the unvarnished portion. In the case of alloys melting at high temperatures, we can allow sufficient gas to pass direct to the furnace by the aid of a by-pass, to keep it at the lowest temperature required, the cut-off thus becoming more efficient. Where ordinary coke fires are used a simple method can be employed which gives very good results. The alloy is made in a small crucible, and when well molten and thoroughly mixed the whole is placed in a hot, larger crucible in place in a bright fire. The outer crucible is filled with charcoal and charcoal powder, to prevent oxidation, and the lid put on; a few pieces of hot coke are placed on top and then the fireplace is filled up with hot ashes; all dampers are closed to shut off as much draught as possible, and the whole is allowed to cool slowly. In this way the size of the structure of the alloy can be increased from ten to twenty times.

Oxidation can usually be prevented by having a good supply of charcoal on top of the alloy. In the case of many alloys whose melting-points are comparatively low, such as the tin antimony series, potassium cyanide is an excellent material to use. In the ordinary way, where very slow cooling is not necessary, some potassium cyanide is melted in a plumbago crucible. The two metals are then added, and when fluid the crucible is shaken, taken out of the furnace and allowed to cool slowly in the open air. When cool, the cyanide is washed away and the alloy taken out.

It is always necessary to thoroughly mix the constituents of an alloy, but in many cases solution and diffusion help materially to form the alloy. For example, to prepare the eutectic alloy of the copper-tin series (1 per cent Cu, 99 per cent Sn) the tin is first melted and the copper added in the form of foil. Although the temperature may only be between  $350^{\circ}$  and  $400^{\circ}$  C., the tin rapidly dissolves the copper, which diffuses uniformly throughout the whole mass. In most cases, if the whole contents of the crucible be liquid, a good shake of the crucible produces a uni-



form alloy. In some cases, however, as for instance the aluminium-bronzes, shaking or even stirring does not always produce a uniform alloy. It is then best to cast the alloy and break it up into small fragments or to granulate it and remelt till the desired result is obtained.

Having obtained our metal or alloy, the next process is to obtain a suitable piece to polish. A convenient size is one from  $\frac{1}{2}$  inch to 1 inch in diameter and about  $\frac{1}{8}$  inch thick. In the case of very hard and brittle metals or alloys, a fragment is broken off of convenient size and a face can be ground down on an emery wheel. When we are dealing with metals which can be cut with a saw and filed, a small plate can be prepared. In the case of bars, rods or rails, much time is saved by using a mechanical saw such as is manufactured by the Millers Falls Manufacturing Company, Mass. With material of a comparatively small size, an American hacksaw and a vise are all that is necessary. It is generally advisable to cut a vertical section, because in some cases the structure is not the same at the top and bottom. This is specially the case with alloys whose constituents, the crystals and ground-mass, differ greatly in density; for example, the alloys of tin and antimony. Having obtained a piece of the requisite size, it is now filed comparatively smooth, using a second cut and then a dead smooth file. This is easily performed by holding the file so that the tag rests on the bench and by drawing the section downwards over its surface. When the section consists of some soft material such as lead or tin, care must be taken to keep the file clean, or particles of metal will cling to the file and tend to tear the surface of the section. This tearing effect tends to produce a new crystallization, and unless the metal thus modified is entirely removed during polishing, we obtain, on etching, a structure similar to that produced by rolling or hammering.\* This point will be referred to later, when change in the solid is considered.

Having filed the specimen comparatively smooth, it is next rubbed over emery paper or cloth of grade o till all traces of file marks are obliterated. Then it is rubbed over paper or cloth of

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\* Campbell: A Note on Crystallization Produced in Solid Metal by Pressure. *Physical Soc.*, London, June, 1900.



grade 00 till the scratches of the previous paper have disappeared. In some cases it is better to grind on a flat, fine turkey-stone, using either water or oil.

The next part of the process consists of rubbing the specimen over sheets of French emery paper, starting with No. 0 through 00, 000 to 0000, always stopping as soon as the scratches of the last paper have been removed, and using as little pressure as possible, especially in the case of soft metals or alloys. The emery paper may be attached to a board of suitable size or may be fixed to a disc which can be revolved by a machine. These papers must be kept absolutely free from grit or the specimen will be spoilt. The last two grades may be moistened with oil.

On leaving the last emery paper, the specimen is polished on broadcloth or chamois leather covered with a little well-washed rouge and well moistened with water. Some workers use an intermediate stage, with diamantine powder. This also has to be washed before using. The operation is finished when the last traces of the emery scratches have been removed. This final polishing must not be carried too far or the specimen begins to polish in relief and an uneven surface is produced. A rouge-wheel saves a great deal of time in this operation.

Several polishing machines have been used to save time. Professor Martens has designed one to polish a number of specimens at the same time. It consists of a block to carry the sections, which are pressed on the grinding and polishing discs of a power-driven revolving wheel. At the Royal Mint, London, Professor Sir Wm. Roberts-Austen uses a series of vertical circular blocks, covered by emery papers of varying grades, and driven by a motor. To polish the specimen, he uses a horizontal rouge-wheel, power-driven and cased in with glass to keep it perfectly free from grits. Stead has designed a small, simple and very efficient machine which can be driven by hand or power. It consists of a horizontal revolving wheel; conical blocks with rings for securing the emery paper, cloth or chamois leather in position; these blocks are simply dropped into the recessed wheel and are carried round by friction. Above is a can to hold either water or the solution to be used; a tap allows the water to drop at a uniform rate upon the center of the polishing block. A shield is used for catching any fluid thrown from the wheel when in motion.



Sauveur has recently devised a machine by which polishing is simplified and the time occupied in the operation greatly shortened. The machine carries four discs revolving in a vertical plane and covered with suitable cloths. The four polishing surfaces are fed automatically from above, and shields are provided to catch any fluid thrown off from the discs when in revolution. He claims that if the samples (of iron) be delivered to the operator after filing them with a smooth file, fifteen and probably more can be prepared in an hour.

The success in polishing depends to a great extent upon the polishing powders. For ordinary work it is found sufficient to use jewelers' rouge which has been carefully washed free from all large particles. We can go further, however, in the preparation of polishing powders and also of emery powders, and obtain an absolutely accurate classification with regard to size. As Le Chatelier points out in his "Notes on the Technology of Microscopic Metallography,"\* this can be performed by the washing method used by Schlöesing for the analysis of kaolins. The carbonate of lime present in tap water causes the formation of lumps of coarse and fine material in the process of levigation. Mr. Schlöesing therefore treats the powders to be classified with water containing some 0.1 per cent nitric acid, which dissolves the carbonate of lime and other salts which may be present, derived either from the water or from the powder treated. When the mixture has settled, the clear liquid is poured off and is replaced by distilled water. The mixture is well stirred and allowed to settle once more. This is repeated till all acid has been removed, when the settling takes a considerably longer time. A little ammonia is now added to help the suspension of the finest particles. The liquid is drawn off at different intervals by means of a syphon, forming a hook at its extremity to prevent the portions which have settled from being disturbed. The different intervals are: fifteen minutes, leaving all the coarse grains unsuitable for polishing; one hour, leaving a powder suitable to start polishing; four hours, when a powder suitable for polishing hard metals, such as iron, is obtained; twenty-four hours, and eight days, when the finest polishing powders are obtained. The last deposit may be precipitated in a few hours

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\* *The Metallographist*, January, 1901.



by adding a little acetic acid. In conducting these operations absolute cleanliness is necessary. The best way to keep the powders is to mix them with soap so as to form a paste. Powdered castile soap is added to the powders whilst still wet, in the proportion of one part of dry soap to ten parts of wet powder. The mixture is melted in a water-bath, well stirred and allowed to cool. Stirring is kept up till the mass commences to thicken, when it is poured into tin tubes like oil-paints, or into paper ones like grease-paints.

Le Chatelier considers the following to be the best powders to use (given in their order of excellence): Alumina derived from the calcination of ammoniacal alum, commercial flour-emery, oxide of chromium derived from the combustion of bichromate of ammonium, and oxide of iron derived from the calcination in air of iron oxalate.

#### DEVELOPMENT OF STRUCTURE

The methods used for the development of the structure are numerous. Etching is the one in most general use. It may be performed with acids, concentrated or dilute, with alkalies or other reagents which attack the specimen. In many cases an electric current has been used with great success. We can also use a combined polishing and etching method, polishing in relief or heat-tinting. The method used depends entirely upon the specimen whose structure we wish to develop. When we wish to follow the changes produced by a certain mechanical or thermal treatment, we must use a constant method for developing the structure; but when we are studying alloys, various reagents should be used for etching; polishing in relief and heat treatment should be employed and the results compared, and in this way the nature of the various constituents can be ascertained.

For carburized irons the combined etching and polishing method of Osmond gives very beautiful results. It consists of rubbing the polished section upon a piece of parchment moistened with an infusion of licorice root, together with a little precipitated calcium sulphate. Instead of these two reagents, a 2 per cent solution of ammonium nitrate may be used. The results are practically the same.



Le Chatelier recommends the use of commercial tincture of iodine diluted with four times its volume of alcohol. The solution is rubbed over the polished specimen with the finger tips several times until a gray film appears. Care must be taken not to etch the specimen too deeply.

Stead describes a tincture of iodine and iodide of potassium (1.25 grains of each in 100 cubic centimeters alcohol) which he uses. He places the specimen on a plate and allows one drop of the tincture per square centimeter. It is left there until the iodine color has disappeared. Wash in water, then in alcohol; dry by means of hot-air blast. If the etching has not gone far enough, the process is repeated.

For irons which show no free ferrite a dilute solution of nitric acid in alcohol (0.5 per cent) is often used. The specimen is dipped into the solution and the etching watched by means of a strong lens. When the etching has proceeded far enough the specimen is placed under the tap, then soaked in alcohol and afterwards dried. If this method is used in the case of ferrite, under high powers the ferrite will be seen to have a granular appearance, due to over-etching. For low-power work, however, it answers well.

For rapidly developing the structure of steels under low powers, a 20 per cent solution of nitric acid is recommended by Osmond. For alloys in general, such as the copper tins, the bearing metals, etc., a dilute solution of nitric acid (1 per cent) will be found to work well; but for alloys which contain much copper or other metal which is comparatively lightly attacked, such as the gun metals, aluminium bronzes, etc., a more or less strong solution must be used.

Heat-tinting, first used by Behrens and Martens, gives very beautiful color effects, which depend upon the formation of films of oxides. The films form at different rates for different constituents. Heycock and Neville have used it with great success, as their papers on the effects of quenching alloys of copper and tin clearly show. Stead has designed a small electrical device for heat-tinting, which can be performed by this means upon the stage of the microscope itself. He has obtained some wonderful results in the case of the alloys of iron and phosphorus.\* Heat-tinting does not, as a rule, show up the granular

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\* *Journal Iron and Steel Institute*, 1900, No. II. *The Metallographist*, Vol. IV (1901), p. 338.



structure, especially that due to differences of orientation; but it is the very best means for showing the different constituents. As it does not remove any metal, the surface always remains flat, and the specimen is less liable to rust than in the case of acids and other reagents.

To preserve specimens from oxidation, etc., the simplest way is to cover the surface with a film of oil. This has the disadvantage, however, that the specimen must be cleaned each time before examination. Le Chatelier recommends that they should be varnished with "Zapon," a solution of gun cotton in amyl acetate. It is transparent and the specimens can be examined under the highest powers.

If we are studying a series of alloys it is often necessary to prepare a large number for fear of overlooking some slight but important change. A preliminary examination embracing the whole series can be made upon one alloy, as pointed out by Le Chatelier. His results were obtained by superposing the two metals so that they mix only in the vicinity of their contact. A vertical section can be taken through the whole mass and the metals examined alloyed in all proportions from one pure metal to the other. This superposition is very difficult to obtain and often takes quite a while to perform successfully; in fact, in some cases, for example where a combination occurs heavier than the heaviest metal, it is impossible to get correct results. In the case of the brasses and the bronzes it yields excellent results.

When dealing with soft metals and alloys it is extremely difficult to obtain a good surface for microscopical examination, because in the process of polishing, the whole of the metal or alloy is not removed; part of it spreads as a thin film over the surface of the specimen and hides the structure beneath. In the case of slowly cooled specimens I believe Le Chatelier has tried to obtain a good surface by inserting a vertical strip of glass reaching almost to the bottom of the crucible, thus dividing the whole into two parts.

In the case of cast metals and alloys, Ewing and Rosenhain\* have obtained some excellent results by casting upon plates

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\* *Royal Society Proc.*, 1899, p. 85. *The Metallographist*, Vol. III (1900), p. 94.

of glass or of highly polished steel. They found, however, that the glass was very liable to break. If this happens before the specimen is solid, or rather whilst there remains any liquid in contact with the glass, we find part of it running through the cracks produced in the glass, thus spoiling the surface.

Mr. H. J. Hannover\* has overcome this difficulty by casting upon mica. He cuts a funnel out of a piece of charcoal, places it on top of a fresh surface of mica, and then pours in his alloy. As this does not always give good results, he casts a small mass of the alloy upon the mica. The alloy thus obtained he places upon another sheet of mica and covers with some potassium cyanide. He then heats it by means of a blowpipe, and when the melting point is reached, places upon it another sheet of mica, previously heated, which he presses down till the alloy is solid. In this way he obtains a very smooth surface on the upper part of the alloy. I have seen this method of casting upon mica in use at the Royal Mint, London, where Roberts-Austen has obtained some very beautiful results.

When casting small ingots of an alloy it is often possible to obtain a clean surface by skimming the metal as it leaves the crucible with a stick of charcoal, retaining any oxides in the pot. On solidification, if this surface be examined, many beautiful and characteristic structures will be met with, and these are often an indication of the internal structure of the alloy.

### MICROSCOPIC EXAMINATION

There are so many papers and books upon the microscope that it will only be necessary here to describe those parts which are more or less peculiar to micro-metallography.

In the first place the stage ought to revolve so that differences in orientation can be observed. It is also very advantageous to have a stage which can be so adjusted that the axis of the microscope is normal to the surface to be examined. This can be effected by means of three leveling screws. Some specimen holders answer this purpose.

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\* *Bulletin de la Société d'Encouragement*, August, 1900. *The Metallographist*, Vol. IV (1901), p. 29.



The next matter of importance is illumination, which is obtained by means of reflectors. For low-power work, using a 1-inch objective, the Sorby-Beck illuminator or reflector is the best (Fig. 2, a). It fits upon the objective. With it oblique and vertical illumination are obtained. To obtain oblique illumination the small mirror, with its face at  $45^\circ$  from the vertical, is turned out of the way. Then the horizontal rays of light are reflected at all angles except vertical upon the surface beneath (provided it is flat and the axis of the microscope is normal to it), and this surface appears dark. Irregularities in the surface will stand out sharply. For vertical illumination the small Sorby mirror is turned into place, and rays of horizontal light falling upon this mirror are reflected vertically upon the surface of the specimen and thence vertically up the tube of the microscope. Horizontal surfaces will therefore appear bright and irregularities will be dark. Vertical illumination for low powers can be simply obtained by a device invented, I believe, by Stead. A small cover-glass is fixed on a glass slide at  $45^\circ$  by wax or cement, and a piece of black cardboard or smoked glass fixed behind it in the vertical. Horizontal rays are reflected just as in the case of the Sorby illuminator; but as they can pass through the cover-glass, this method is rather better for powers below 1 inch, for the Sorby mirror, being attached to the objective, is comparatively far from the specimen.

For high-power work, using higher powers than 1 inch, the Beck illuminator answers very well. In Fig. 2 the short cylinder contains a small disc of glass; light enters through the circular opening at the side. The illuminator is fixed between the objective and the tube of the microscope. The disc of glass is set at an angle of  $45^\circ$ , and so light is reflected down the tube, through the objective and onto the specimen. This gives vertical illumination, of course. When working with several powers, a multiple nose-piece or a Zeiss objective slide saves much time.

For high powers prism illuminators are also used; these fit between the objective and the microscope tube and work like the Beck. Stead recommends that made by Nachet. He uses the Sorby-Beck with a 1-inch objective, a Nachet prism with the  $\frac{1}{4}$  or  $\frac{1}{8}$  inch, and the Beck with the  $\frac{1}{8}$  inch or  $\frac{1}{16}$  inch oil immersion.

For illumination, an incandescent Welsbach light answers



well for low powers, and for high powers an arc lamp gives the best results. Bull's-eye condensers are generally necessary for all work. A small acetylene lamp has found favor with many workers.

For photographing the object, two methods can be used. In the first case the camera and microscope are vertical, whereas in the second they are horizontal. They both have their advantages. When vertical, it is comparatively an easy matter to place your object; when horizontal, it has to be firmly fixed. But with the vertical method the bellows of the camera must be restricted as to length unless you wish to use a step-ladder, or have your dark slide-holder and focussing glass in the room above; whereas, in the horizontal method, the bellows can be of great length and the magnification proportionately increased.

It is really a matter of taste which to use. Stead uses the vertical method, Roberts-Austen the horizontal.

Fig. 1 shows the apparatus used in Professor Howe's laboratory; it is manufactured by the Bausch & Lomb Optical Company and can be used in either position.

The method used by Le Chatelier is fully described in January impression of *The Metallographist*, 1901, page 10.

To any one interested in the details of practical metallography a paper by Stead will prove of great value. It was a lecture delivered to the Cleveland Institution of Engineers in February, 1900. It is reprinted in *The Metallographist* for July, 1900. Papers by Le Chatelier,\* Charpy,† Osmond,‡ Sauveur,§ and many others, go into the various details so thoroughly that any one can learn with a little patience the art and practice of the science. To those whose time is too fully employed to allow them to read all papers on the subject in the original, Sauveur's journal, *The Metallographist*, will give all that is necessary to follow the subject thoroughly.

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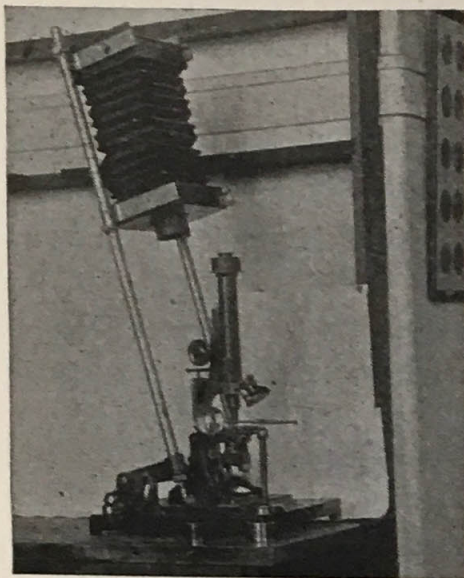
\* *Revue Générale des Sciences*, January, 1897. *The Metallographist*, Vol. IV, No. 1.

† *Bulletin de la Société d'Encouragement*, March, 1897. *The Metallographist*, Vol. I (1898), p. 87.

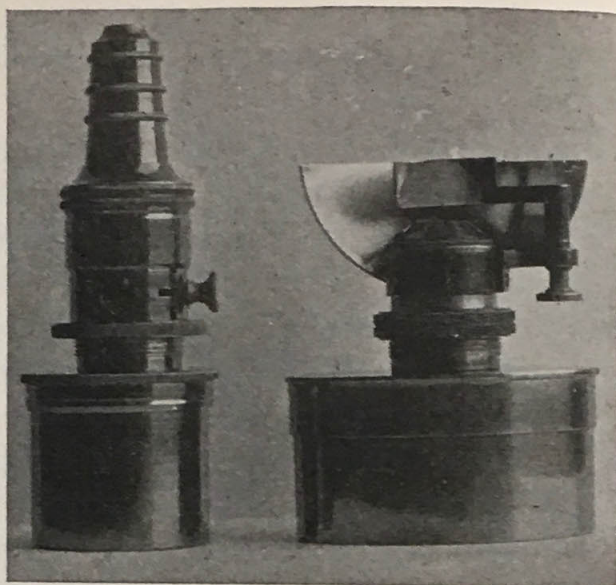
‡ *Rapports de la Commission des Méthodes d'Essai*, Vol. II. *The Metallographist*, Vol. III.

§ *The Metallographist*, Vol. IV, No. 4.



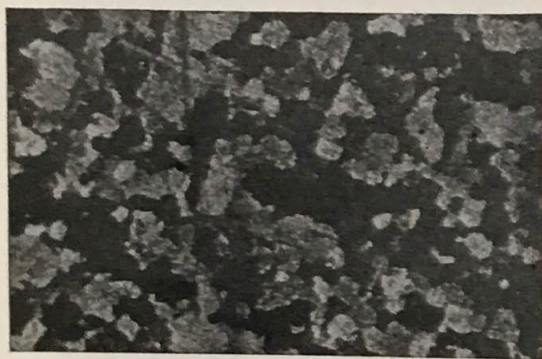


1.



2.

a.



3.

x 33. v.

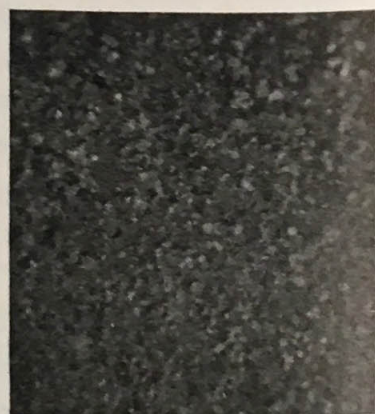


4.

x 33. v.

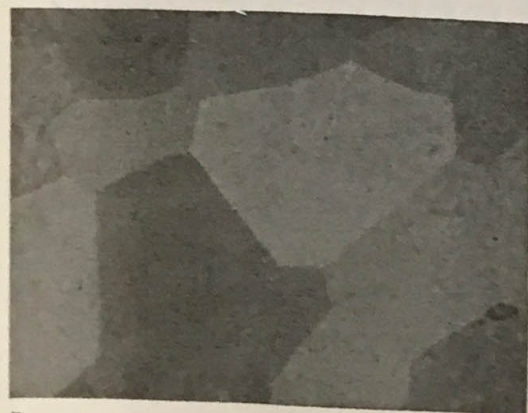


5.



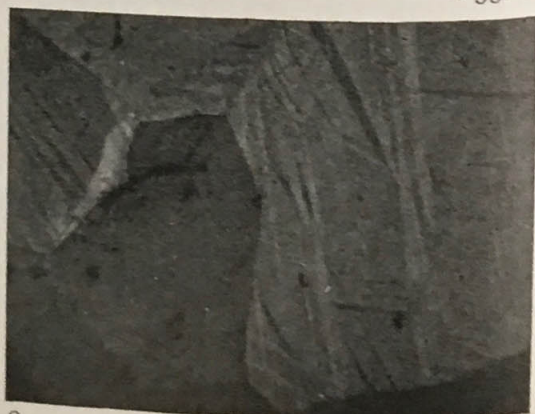
6.

x 33. o.



7.

x 33. o.



8.

x 33. o.



## THE CRYSTALLINE STRUCTURE OF METALS

In the *Annales des Mines* for January, 1900,\* Osmond published a paper on the crystallography of iron, with some very beautiful illustrations. He concludes that iron in its three states crystallizes in the cubic system, but whereas, *Gamma* iron crystallizes generally in octohedra, *Beta* and *Alpha* iron both crystallize out in cubes. In a later paper written in conjunction with G. Cartaud,† he confirms his previous results by causing the three allotropic varieties of iron to crystallize out as pure as possible, and at that temperature in which each variety is normally stable, the conditions being such as to allow free development of the crystals, the external forms remaining unaltered by the internal changes which might occur during subsequent cooling. The experiments were conducted in a porcelain tube, internally varnished, placed in a Mermet furnace. A Le Chatelier pyrometer was used to ascertain the temperature, which could be regulated by the gas supply.

*Alpha* and *Beta* irons were prepared by Pélégot's method,‡ which consists in reducing ferrous chloride by hydrogen; whilst *Gamma* iron was prepared by the method used by Poumarède,§ in which zinc vapor replaces the hydrogen for the reduction of the ferrous chloride. Since the surrounding atmosphere has some influence on the results obtained, some *Gamma* iron was prepared by the Pélégot method and some *Alpha* and *Beta* iron by the Poumarède and the results compared. The paper is full of beautiful photographs and drawings, showing the various forms of crystals obtained. In many cases the crystals shown are identical in form with those prepared at South Kensington by Dr. E. Ball, who distilled galena in a porcelain tube *in vacuo*.

Ewing and Rosenhain|| have developed the crystalline structure of many of the softer metals by casting on glass or polished steel. On examining the surfaces thus prepared various crystalline forms can be clearly seen.

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\* Also *The Metallographist*, Vol. III (1900), p. 181.

† *Annales des Mines*, August, 1900. *The Metallographist*, Vol. IV (1901), p. 119.

‡ *Comptes Rendus*, Vol. XIX, p. 670, 1844.

§ *Comptes Rendus*, Vol. XXIX, p. 518, 1849.

|| *Proc. Royal Soc.*, 1899. *The Metallographist*, Vol. III (1900), p. 94.



Another way of examining the structure of some metals and of demonstrating their methods of growth is by allowing part of the metal to solidify and then removing the liquid portion. The beautiful crystalline structure of bismuth is brought out in this way. If pure lead be melted in a crucible and allowed to solidify round the outside, by pouring out the whole of the liquid contents a beautiful dendritic structure is revealed. Many beautiful specimens prepared in the laboratory of Dr. Percy are now in the collection at the Royal School of Mines, London.

This dendritic growth of crystals in metals is of very common occurrence. In the case of iron, dendritic crystals often occur in the cavities existing in that portion of the large ingots which solidifies last. Professor Tschernoff has quite a large collection, some of which are about 2 feet long.\* On examining the surfaces of small ingots of most metals (and many alloys) we see many good examples. Lead and tin show them. They stand out above the surface of the ingot because they have grown at the surface, and as the metal solidifies the mass contracts more or less, the molten part sinks and they are left standing out in relief. Fig. 9 shows such dendrites of cadmium, which have the appearance of six-rayed stars, symmetrically arranged about a hexagonal axis. From each axis grows a series of parallel branches, making an angle of  $60^\circ$  with that axis, and therefore parallel to the next adjacent axis. Between each pair of axes there is seen the beginning of another axis, thus adding to the hexagonal symmetry.

Fig. 10 shows the surface-structure of a bar of aluminium. Two intersecting dendrites are seen. In each only one quadrant has developed. We have two axes at  $90^\circ$ , and from each grow parallel branches normal to their own axis, but parallel to the adjacent axis. From these branches grow leaf-like grains, thus forming a true skeleton crystal. The whole shows a decided cubic character. The cubic structure of aluminium can be clearly shown by casting a small ingot of the metal. When half solid, the ingot is tilted and the liquid part is allowed to drain off. The rough surface thus exposed is seen to be divided up into many areas; each area is made up of a number of small cubes similarly orientated, whilst each cube is made up of row upon row of

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\* *The Metallographist*, Vol. III, No. 3, p. 199.



minute grains. The whole structure resembles a surface of silicon steel ( $4\frac{1}{2}$  per cent) in Stead's Crystalline Structure of Iron and Steel.\*

Copper, platinum, gold and silver show this dendritic structure on their surfaces when suitably cooled. Fig. 11 shows the structure met with on the surface of a cupellation button of silver. Only the centre of the skeleton crystal is shown, but the two main axes can be seen at right angles to each other; from each grow out, normally, parallel rows or branches composed of grains. The nearer to the centre of crystallization, the smaller are the grains. In Fig. 12 a smaller skeleton crystal is shown. It is similar to the large ones, but is stunted in growth. It shows the curious growth at the ends of the axes where they merge into the surrounding surface. Although these skeleton crystals are fairly common, they do not occur on all silver buttons. The normal structure of the surface of a cupellation button of silver is shown in Fig. 13. It consists of grains with straight boundaries. This structure is probably the result of contraction on solidification, because, in places, the lines are seen to cut across patches of similar orientation, i.e., crystals. The true area of each grain (crystal) is seen by its orientation, and the lines of fissure generally coincide with the true grain (crystal) boundary because these will be the lines of weakness. In some cases, however, they take a direction of their own. This cooling structure is probably similar to that met with in many basalts. The surface structure of a silver ingot is seen in Fig. 14. There are several large grains or crystals with irregular boundaries but distinct orientation, each made up of secondary grains, similarly arranged, causing that orientation.

In the case of bismuth, Fig. 15, we see that the dendrites are inclined to assume a definite crystalline form; and this is exactly what we should expect when we remember the definite forms seen on a crust from a crucible of bismuth. The stars of antimony are too well known to require any description here.

When certain metals are cast, dendrites are formed by the shrinking of the liquid part during solidification, thus leaving them standing above the surface. A similar thing occurs when

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\* *Iron and Steel Institute*, May, 1898. *The Metallographist*, Vol. I (1898), p. 289.



many alloys are cast. We may have dendrites of a metal left in relief, due to the shrinking of the eutectic or portion of the alloy which solidifies last; or we may have definite crystals, generally of a compound, standing up above the surface. These surface structure, as we said before, are often very reliable indications of the internal structure of the alloy. They may also be very misleading, however, as in the case of the bronzes between 70 per cent and 95 per cent of copper.

In his paper on the "Crystalline Structure of Iron and Steel,"\* Stead points out that the same law which determines the size of the individual crystals formed when hot saturated solutions of chemical salts are cooled quickly or slowly holds good when metals solidify from the liquid state. If a metal be cooled very slowly past its freezing point, its structure will be comparatively large; if it be cooled rapidly past its freezing point, the result will be a much finer or more minute structure. In cooling slowly, crystals start to grow from a comparatively few points only, and the resulting structure is large; in rapid cooling, the points of initial crystallization are numerous, and the structure on complete solidification is small. When a crystal starts from a point it grows in all directions till interfered with by the growth of adjacent crystals. As the crystals seldom grow so that their crystallographic axes lie in the same directions, we find on solidification that each has a different orientation. This difference in orientation is beautifully seen if we take ingots of lead, tin, cadmium, zinc, etc., and lightly etch their surfaces. The effect of etching is to take off the surface of the metal, but not as a film of uniform thickness. The acid has not the same effect on each crystal, but removes more of one than of another, the amount depending upon the position of the crystal with relation to the surface which is being etched. The acid follows the track of the secondary crystals, or rather grains, and so we have a step-like surface eroded, which resembles, more or less, a broken surface of galena. Thus, light is more or less reflected from each crystal, depending on the angle at which the step-like surface is inclined.

The size of the crystals of an ingot of metal and, to a great

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\* *Iron and Steel Institute*, May, 1898. *The Metallographist*, Vol. I (1898), p. 289.



degree, their shape depend upon the mould in which the metal is cast. Thus tin, when cast in a stone mould, gives quite a different appearance to what it does when cast in iron; the difference, of course, being due to the difference in rate of cooling. Again, the size of grain or crystals depends upon the purity of the metal used. If pure and impure tin be cast in the same mould, in the case of the impure tin the crystals will be comparatively long and slender, their length pointing towards the axis of the ingot.

The fractures of ingots in general reveal a columnar structure. If an ingot of zinc be broken, elongated crystals with irregular faces are seen to have grown from the surface next to the mould perpendicularly towards the centre of the ingot. If a section of an ingot of lead or tin be cut vertically, polished and etched, a similar structure is seen. If ingots of lead are broken hot, this columnar structure is beautifully revealed, whilst cast cylindrical rods of most metals show a radial structure, due to the same cause. On fracturing a cast rod of antimony the structure is that of numerous elongated pyramids with their apices meeting at the axis of the rod, their bases forming the cylindrical surface of the rod.

### THE EFFECTS OF STRAIN

The effects of strain upon metals have been well explained and illustrated microscopically by Ewing and Rosenhain.\* Charpy† has illustrated the effects of strain and also of annealing upon the brasses, etc.; Roberts-Austen‡ upon steel rails and gun tubes.

Ewing and Rosenhain state that no effects of strain can be detected under the microscope within the elastic limit; but as soon as this is passed, fine dark lines are seen on the faces of the grains. They are more or less straight, are parallel in each grain, but are differently orientated in different grains. The first lines

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\* Bakerian Lecture, May 18, 1899, *Phil. Trans. Royal Soc.*, CXCI, p. 353. *The Metallographist*, Vol. III (1900), p. 94.

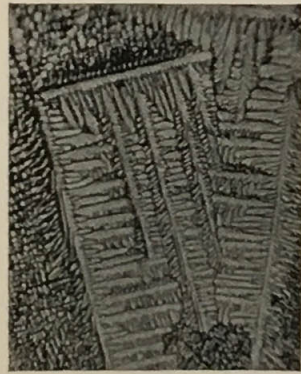
† *Bulletin de la Société d'Encouragement*.

‡ *Journal Iron and Steel Institute*, No. II, 1898; No. II, 1900. *The Metallographist*, Vol. II (1899), p. 125.

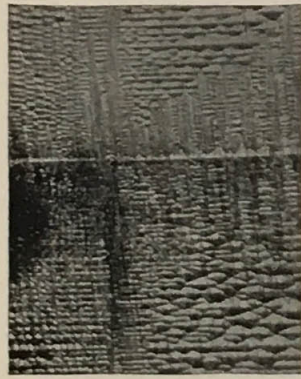




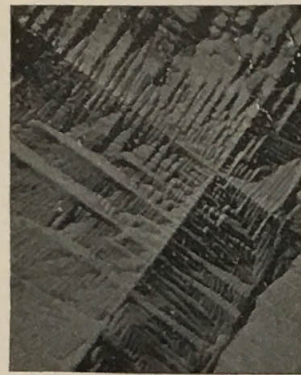
9. x 33. v.



10. x 33. v.



11. x 33. v.



12. x 33. v.



13. x 16. v.



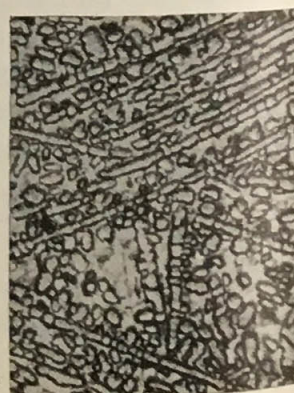
14. x 33. v.



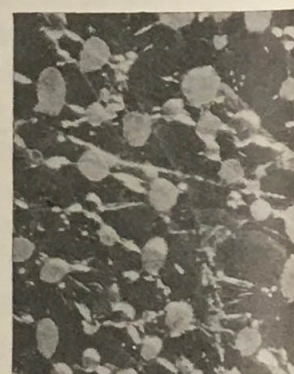
15. x 33. o.



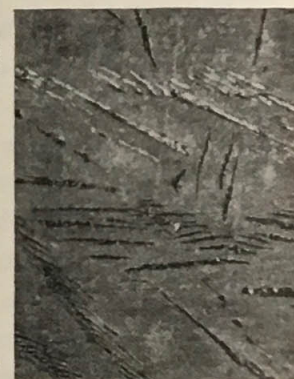
16. x 33. v.



17. x 33. v.



18. x 33. o.



19. x 33. o.



20. x 33. v.



that appear are those approximately transverse to the line of force; but as the force increases, other systems of lines appear on other grains, and these lines are inclined to the direction of straining. As the straining increases some of the grains begin to show one, two, three and more systems of parallel lines, thus giving the grain the appearance of Schillerization. If the surface of a bar of bismuth, which has been strained till it broke, be examined, the lines or slip-bands are clearly seen on each side of the fissure. Several parallel groups will appear in view. Fig. 6 in Dr. Mathew's paper showed some of these slip-bands in tin. It shows the strained surface of an ingot of pure tin, whilst Fig. 5 of the same paper showed lines of strain set up in a section of tin which had been hammered and then annealed. Similar slip-bands in crystals of cadmium are shown in Fig. 8. The cry of tin, the crackling of zinc, etc., are due entirely to the formation of these slip-bands.

It was mentioned above that when a soft metal, such as lead or tin, was cut with a file, particles tend to cling to the file and tear the surface of the metal. A similar thing happens with nearly all the softer alloys. The immediate effect is not noticed; but on etching, long parallel lines of irregular grains or crystals are seen to have been developed. Fig. 3 shows such lines developed in tin. When a soft metal or alloy is cut with a saw, a similar crystallization is set up, and unless the metal thus modified is removed during subsequent treatment, this crystallization appears along with the original structure on etching. Fig. 4 shows a section of slowly cooled tin which has been cut with a saw; a finer crystallization is seen together with the larger structure, due to the original cooling. If etching be prolonged the finer structure is obliterated, showing that it is only on the surface.

Now, when a piece of metal is hammered or rolled the crystals become lengthened in the direction in which it has become drawn out, and shortened in the direction of contraction. In the case of copper, iron and other of the tougher metals, this attenuating stage is of comparatively long duration; but in the case of the softer metals it is short and the crystals or grains break up to form a new and minute crystallization. Fig. 2 of Dr. Mathews' paper shows the appearance of hammered tin. The structure is roughly granular and no trace of the original



slow-cooling structure can be seen as in Fig. 4. Rolled tin has a similar structure, and it is curious to note that the thinner the piece is rolled the more distinct become the individual crystals. At a certain point they no longer split up, but become attenuated as if plastic. The structure of rolled cadmium is shown in Fig. 6.

### THE EFFECT OF HEAT-TREATMENT

It is well-known that annealing tends to relieve the strain in a metal or alloy. In many metals it does more than this; it causes the growth of crystals or grains. Fig. 1 of Dr. Mathews' paper shows a bar of tin rolled out to about 3.5 millimeters thick. The right-hand pair show the structure of the metal just as it comes from the rolls. The left-hand pair have been annealed for ten days upon a hot-plate at not more than  $180^{\circ}$  C. The size is slightly reduced. The difference between the unannealed and the annealed specimens is striking, to say the least. The orientation in the left-hand pair is marked and shows the boundaries between the various crystals. Figs. 3 and 4 of the same paper show hammered tin unannealed and annealed at about  $180^{\circ}$  C. for seven days on a hot-plate (not ten days as stated). Again the difference between the two specimens is striking. In Fig. 4 it will be noticed that the new crystals tend towards straight regular boundaries and in section are more or less perfect hexagons.

Fig. 5 shows five pairs of strips of cadmium rolled out from a rod. Starting from the right, the first pair have a thickness of 0.075 inch, the second of 0.049 inch, the third of 0.036 inch, the fourth of 0.024 inch, whilst the last and thinnest pair are only 0.018 inch in thickness. They were rolled out from a rod having a circular section  $\frac{5}{16}$  inch in diameter. Their length is  $2\frac{5}{8}$  inches and breadth 0.4 to 0.45 inch.

The right-hand strip of each pair has been annealed for seven days in an air bath at a temperature under  $180^{\circ}$  C. Etching in all cases was with nitric acid dilute. After rolling, the thickest pair (No. 1) were found to have the coarsest structure and this is shown in Fig. 6. After annealing, although tolerably large crystals were found in all of the strips, the thickest strip was found to have the coarsest average grain. Fig. 7 shows the crystals developed in No. 3 by annealing. The definite bound-



aries and distinct orientation are to be noted. Those developed in No. 1 were so large that a single crystal would cover the whole figure at the same magnification, whilst those developed in the thinnest strip were proportionately smaller. Fig. 8 shows some of the smaller grains or crystals of No. 1, photographed after the strip had been bent. The strain produced by bending has set up the series of slip-bands. Five distinct parallel series can be seen in the largest grain on the left.

An exactly similar experiment was performed with tin. The annealing was carried on for five days on a hot-plate and the results were identical with those of the cadmium. In both cadmium and tin, annealing produces a marked brittleness, and when the strips are distorted they cry.

If ordinary rolled sheet lead be etched and examined, a structure similar to Fig. 6 will be met with. Etching is best performed with acetic acid for a long period. Anneal it for a week at a temperature of only  $200^{\circ}$  C., and a coarse, very irregular crystalline structure will be developed. The crystals will be of all sizes and shapes; in many, distinct twinning will be observed. In all cases the difference in orientation will be marked. In one case a crystal quite  $\frac{1}{2}$  inch long by  $\frac{1}{2}$  inch in breadth was grown in five days by the author. The temperature was only  $180^{\circ}$  C. and the thickness of the sheet of lead 1.75 millimeters.

Take zinc and roll it out into strips; it will take a fairly high power to reveal its structure. Anneal it as before shown and you will find the crystals have grown so large that some two dozen will be all that are able to occupy your strip. The rolled zinc will be exceedingly tough, but after annealing it will crackle on bending ever so slightly and will break if further bent, for annealing has made it extremely brittle. On slightly bending it and then examining it under the microscope, it will be seen that each grain is traversed by parallel systems of slip-bands, which develop into cracks if the bending be carried to any considerable extent.

If the surface of electrolytically deposited copper be examined, a coarse crystalline structure is seen. On parts of the sheet it will often be found that the rate of deposition has been greater than the surrounding surface and a crystalline knob of copper stands out above the level of the rest of the sheet. If a section



be made through one of these knobs, and the surface be polished and etched, the true crystalline structure is shown up. It consists of comparatively large, irregular crystals, each possessing a definite orientation. If this copper be rolled out, the large crystals are broken up and a much finer crystallization is set up, still composed of crystals with irregular boundaries. If copper foil be examined after its repeated annealing and rolling, or rolling alone, it is seen to be composed of very small grains. The foil possesses a marked amount of spring. No amount of etching will show any orientation, and the structure seems to be composed entirely of oval grains, pointing, of course, in the direction of rolling. It is similar to that of sheet-copper which has been rolled out from an ingot. Anneal it for a moment at a straw heat and it becomes quite limp with no trace of spring left. On examination the fine crystalline structure has disappeared and a large mottled structure is now seen. Thus there is an intimate relation between the fine rounded or oval-grained structure and elasticity of the copper foil.

Electro-nickel when polished and etched shows a reniform structure where it has formed a knob-like mass. Numerous distinct layers can be seen, but, so far, no distinctly crystalline structure can be seen. The structure of iron appears to be similar to that of nickel, that is, it consists of layer upon layer like an agate. The effects of annealing and rolling electro-nickel and iron have not been examined as yet by the author.

The growth of crystals in the solid due to annealing has been shown over and over again. Roberts-Austen, in his last Cantor-Lectures, delivered to the Society of Arts, London, exhibited three photomicrographs showing the growth of crystals in pure gold. The first showed two plates of pure gold pressed together. The squeezing was so great that the crystals had been strained. Without strain, it is thought that no welding will take place. The second view showed the plates after annealing at  $750^{\circ}$  C. and slowly cooling. The junction between the two plates could be distinguished, but in several places crystals had grown across the junction. A third view showed the same after long annealing and the line of junction had entirely disappeared. If a section be taken across a weld in a steel rail or bar which has been joined by the Goldschmid welding process, no line of junction can be seen.

## ALLOYS

Under the heading of "Alloys as Solutions," Dr. Mathews points out that certain metals when liquid at ordinary temperatures do not mix or alloy, and on standing separate into two layers, the heavier at the base.

The following are some of Dr. Alder Wright's figures, obtained by taking each of the five pairs well mixed by stirring, and then allowing them to stand molten for some hours at  $800^{\circ}\text{C}.$ ; separation took place; two liquid alloys were formed; these were quenched and each analyzed.

In the case of zinc and lead, the heavier alloy contained 1.3 per cent Zn; the lighter 1.57 per cent of Pb.

In the case of zinc and bismuth, the heavier alloy contained 14.28 per cent Zn; the lighter 2.32 per cent of Bi.

In the case of aluminium and lead, the heavier alloy contained 0.07 per cent Al; the lighter 1.91 per cent of Pb.

In the case of aluminium and bismuth the heavier alloy contained 0.28 per cent Al; the lighter 2.02 per cent Bi.

In the case of aluminium and cadmium, the heavier alloy contained 0.22 per cent Al; the lighter 3.39 per cent Cd.

If we allow our molten metal to cool down very slowly till it solidifies, and then examine the two alloys thus formed, in the case of zinc and lead or aluminium and lead, and most probably also of aluminium and bismuth or aluminium and cadmium, we get the same results. Each alloy is a solid solution of one metal in the other; no crystals but those of the metal in excess can be seen. Take the aluminium-cadmium pair. The structure of the upper part is that of aluminium, and all of the cadmium it contains is in solid solution. The structure of the base is that of cadmium, and all the aluminium in it is in solid solution. In the case of the lower alloy of the lead-aluminium pair, it is probable that the lead throws all of the aluminium out of solution before solidification, because, according to Heycock and Neville, the freezing point is not depressed at all.

With the zinc-bismuth pair matters are different. The upper alloy shows zinc containing all the bismuth in solid solution, but the lower one shows a fine crop of crystals in the groundmass, which is probably a eutectic of bismuth and zinc, as shown in Fig. 16. Thus between bismuth and about 13 per cent Zn, 87 per



cent bismuth, we get one layer in each alloy we make. Between these points we have a simple series of alloys, whose freezing-point curve consists of two branches inclined to each other and meeting at the eutectic point 3.5 per cent Zn at about  $240^{\circ}$  C.\*

If either of the five pairs of metals above be melted and a sufficient proportion of either tin, silver, antimony or copper be added, no separation will take place; but if the quantity of the "solvent" metal is less than a certain amount, the alloy separates into two layers, each layer consisting of a ternary alloy of the three metals. Fig. 17 shows a case where sufficient of the "solvent" metal was added and a single ternary alloy was formed. The alloy consists of equal weights of zinc, bismuth and tin. The figure shows long dendrites, which closely resemble those of zinc in zinc-tin alloys, set in a groundmass, probably a triple eutectic of BiZnSn.

In many text-books the amounts which each of the above pairs of metals retain of the other when solid varies widely. In some cases we are told that aluminium mixes freely with both bismuth and cadmium. The reason for this lies in the method of cooling. If instead of cooling slowly we cast or chill our alloy, directly after stirring, we don't allow our emulsion to settle, and in consequence *globules* of the heavier are found in the lighter alloy. Fig. 18 shows the surface of aluminium containing such globules. Aluminium was melted with some Bi and Cd. When thoroughly melted the pot was shaken and the alloy poured into a cold iron mould. In consequence of the rapid cooling, separation could not take place, and scattered throughout our aluminium appear shots or globules of the bismuth-cadmium alloy. When slowly cooled perfect separation took place.

## BINARY ALLOYS

Charpy† has published one of the most complete papers on the microstructure of binary alloys. He classes them according to their curves of fusibility, following Le Chatelier.

\* Gautier: *Bulletin de la Société d'Encouragement*, Vol. I, 1896, p. 1293. Roberts-Austen; "Introduction to Metallurgy," plate pp. 114-115.

† *Bulletin de la Société d'Encouragement*, March, 1897. *The Metallographist*, Vol. I (1898), p. 87.



*Group I.* — Two branches of curves starting from the melting-points of the two pure metals and meeting at a point corresponding to the eutectic alloy; this curve is obtained when the metals form neither definite compounds nor isomorphous mixtures.

*Group II.* — Three branches of curves, two of them starting from the melting-points of the pure metals and a third exhibiting a maximum and crossing the two former in two points corresponding to two eutectics; it is the case of two metals forming a definite combination.

*Group III.* — The curve of fusibility is continuous and unites the melting-points of the two metals forming isomorphous mixtures.

Fig. 14 in Dr. Mathews' paper shows types of these curves. Group I is represented by the AgCu curve, Group II by the SbCu curve, and Group III by the AuAg curve.

To Group I belong all those alloys in which the excess of either metal crystallizes out in a eutectic which is a mechanical mixture of the two metals.

Lead and antimony.  
Tin and zinc.  
Tin and lead.  
Tin and bismuth.

Lead and silver.  
Zinc and aluminium.  
Copper and silver.  
Copper and gold.

Dr. Mathews' explanation of Fig. 18 of his paper holds good in the case of all the above alloys, the only difference being that in some cases the compositions *b* and *d* represent comparatively large figures, as for example in the case of copper and silver or copper and gold; in some cases *b* and *d* represent compositions of solid solutions infinitely dilute; that is, *b* and *d* are numerically very small, as in the case of tin and zinc, etc.

We can form a sub-group in which the excess of one metal crystallizes out in a eutectic which is a solid solution of this metal in the second.

Aluminium and tin.

In Group II we get two divisions for each pair of metals alloyed, each division being comparable with Group I, thus:

(1) The excess of the first metal or the compound crystallizes out in a eutectic which is a mechanical mixture of this metal and the compound.



(2) The excess of the second metal or the compound crystallizes out in the second eutectic, which is a mechanical mixture of the second metal and the compound.

Copper and antimony.  
Tin and nickel.

A sub-group might be formed in which the eutectics are solid solutions of the compound in either the first or the second metals.

Antimony and aluminium.

Thus we get two divisions, each of which is comparable with the sub-group of Group I. In the case of antimony and aluminium we have the compound  $\text{SbAl}$  crystallizing out in a eutectic which is a solid solution of Sb in Al, or in a eutectic which is a solid solution of Al in Sb.

In Group III no eutectic in the true sense of the word exists. The examples of this group are:

Bismuth and antimony.  
Silver and gold.

When an alloy of gold and silver commences to solidify, dendrites comparatively rich in gold begin to form. The mother-liquor is enriched in silver. As the temperature falls, the dendrites continue to grow larger and larger, but as each coating of metal is less rich in gold than the one previous, and as diffusion (in the case of slow cooling) produces a uniformity of composition or an equilibrium in the dendrites, these latter become richer and richer in silver, till at the point where the whole alloy becomes solid they have the composition of the original alloy. Of course, if equilibrium is not established we get mixed crystals, or in other words the dendrites are richer in gold at the centre than at the outside.

A fourth group might be added — where a compound is formed and we get two divisions:

(1) An excess of the first metal or the compound crystallizing out in the eutectic of this metal and the compound.

(2) An isomorphous mixture of the compound and the second metal.

To this group would belong antimony and silver and perhaps

tin and silver. Thus this group is really a combination of Groups I and III.

Charpy groups together alloys with abnormal curves of fusibility. Amongst them are:

Copper and tin.  
Copper and zinc.

Antimony and tin.  
Zinc and silver.

## TIN AND ZINC

The cooling curve of the series (Fig. 33) is composed of two inclined branches meeting at 8 per cent zinc. The horizontal branch corresponding to the freezing of the eutectic occurs at  $205^{\circ}\text{C}$ ., and extends from almost pure tin to almost pure zinc; in other words, as soon as a very small quantity of one metal is added to the other a eutectic is formed, for the concentration of the solid solutions formed is very dilute.

Then between tin and the eutectic alloy containing 8 per cent zinc we find grains, and then dendrites of tin set in an increasing groundmass of eutectic. The solidification of the tin is marked by the break  $BC$  in Fig. 33, whilst that of the eutectic is denoted by the break  $bc$ . Below  $bc$  the alloys are solid; between  $BCb$  we have a mixture of solid grains or dendrites of tin in a liquid whose composition will depend upon the temperature.

Between zinc and the eutectic alloy we find grains, dendrites, and then long, attenuated, lath-like bodies of zinc surrounded by increasing amounts of eutectic. The solidification of the zinc is marked by the break  $AC$ , whilst that of the eutectic is denoted by  $aC$ . Then, as before, below  $aC$  all the alloys are solid. When an alloy cools down from the liquid state, it remains entirely liquid till its temperature reaches that of the curve  $AC$ . For example, take the alloy containing 80 per cent zinc. At  $390^{\circ}\text{C}$ . the first grains of zinc begin to form and the mother-liquor is enriched with regard to tin. As the temperature falls, the zinc grains grow and the mother-liquor becomes richer and richer in tin. Thus at  $350^{\circ}\text{C}$ . it will contain about 47 per cent of tin; at  $300^{\circ}\text{C}$ . about 72 per cent; whilst at just above  $205^{\circ}\text{C}$ . it contains about 92 per cent. At  $205^{\circ}\text{C}$ ., that is, the temperature  $aCb$ , the mother-liquor solidifies and the temperature remains constant till the whole mass is solid; then the temperature falls normally



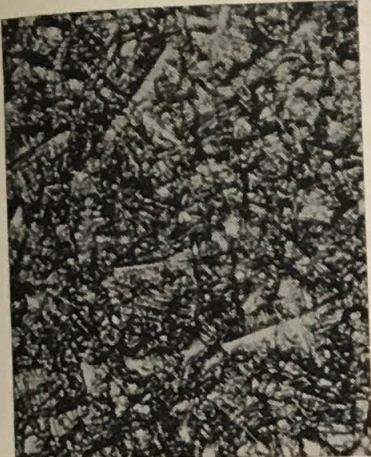
in the solid mass. Fig. 19 shows an alloy containing 20 per cent zinc, 80 per cent tin. The long, lath-like grains of zinc are seen with their distinct tendency towards parallel growth. Fig. 17 would well illustrate the appearance of an alloy containing between 60 and 70 per cent Zn.

## LEAD AND TIN

The cooling curve for these two metals, taken from the Report of the Alloys Research Committee (London), is shown in Fig. 33. It consists of two inclined branches meeting at the eutectic point, 68 per cent tin, 32 per cent lead. The horizontal branch corresponding to the solidification of the eutectic occurs at  $180^{\circ}$  C. and probably extends further than shown, for it would appear in this case also that the concentration of the solid solutions formed was very minute. The meaning of this curve is the same as that of the tin-zinc series. Alloys containing more than 68 per cent Sn remain liquid until their temperature reaches *C B*. At that point pure tin or almost pure tin crystallizes out of the mother-liquor and continues to crystallize out till the mother-liquor reaches the composition of 68 per cent Sn, which it does at  $180^{\circ}$  C. At that point it solidifies as a eutectic of alternate laminae more or less curved of lead and tin, without any fall in temperature. When solidification is complete the temperature falls normally. Alloys containing less than 68 per cent Sn remain liquid till they reach the temperature of branch *A B*, when lead commences to crystallize out, enriching the mother-liquor in tin. Crystallization continues until the mother-liquor has a composition of 68 per cent Sn at  $180^{\circ}$  C., when it solidifies as before.

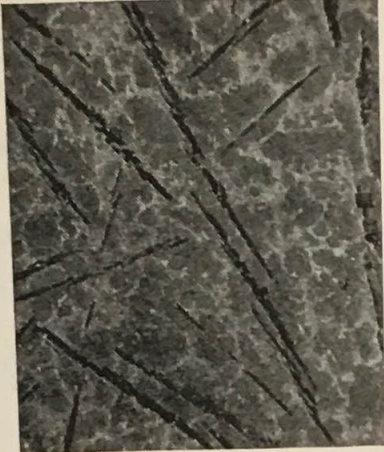
The surface structures of these alloys when cast, and also when slowly cooled, give a good indication of their internal structure. Fig. 20 shows the surface structure of the alloy 15 per cent Pb; 85 per cent Sn when cast. The large dendrites of tin, differently orientated, are seen standing out above the surface. The eutectic, in cooling and solidifying, has shrunk and sunk beneath the level of the dendrites, which thus stand out in relief. Fig. 21 shows the surface of an alloy from the other end of the series. It contains Sn, 26 per cent; Pb, 74 per cent. It is taken from a small ingot. In this case the dendrites are those of lead,





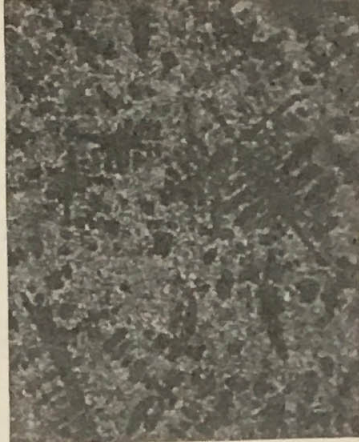
21.

x 56. o.



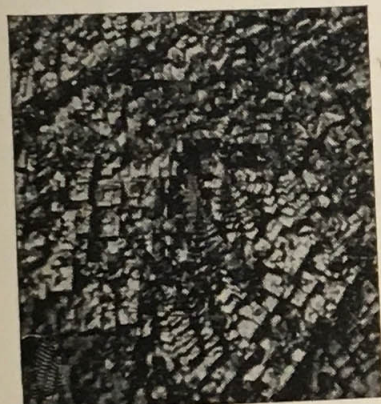
22.

x 33. o.



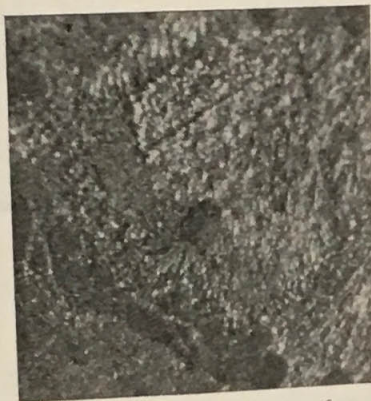
23.

x 33. v.



24.

x 30. v.



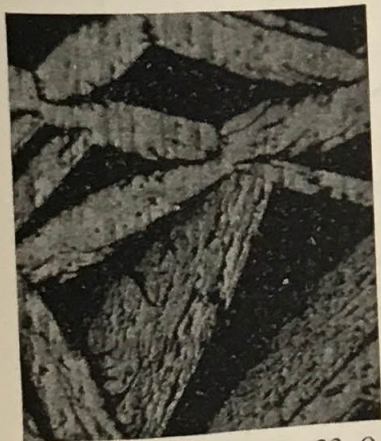
25.

x 56. o.



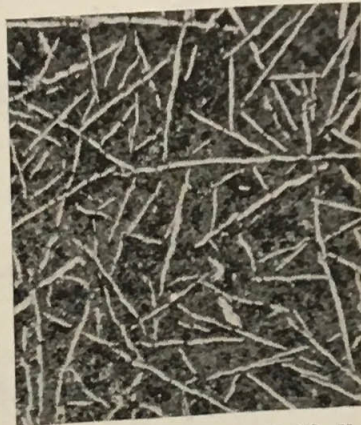
26.

x 33. v.



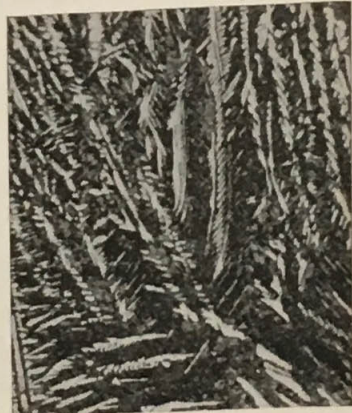
27.

x 33. o.



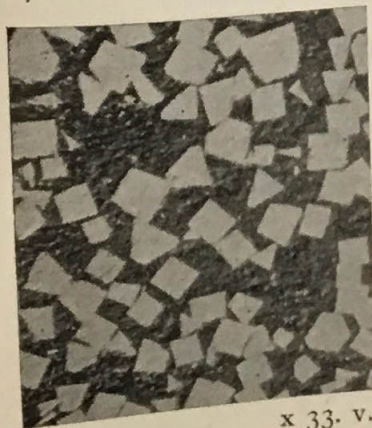
28.

x 33. v.



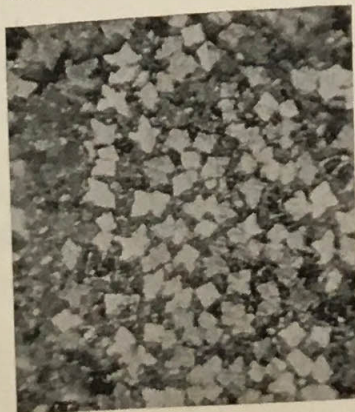
29.

x 16. v.



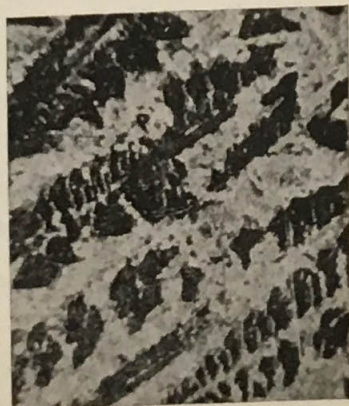
30.

x 33. v.



31.

x 33. v.



32.

x 16. o.



which stand out above the level of the eutectic. The difference in size of the dendrites of tin and lead is great.

Fig. 22 shows the effect of copper upon common solder. Five per cent of copper was added to the solder (2 Pb:1 Sn)

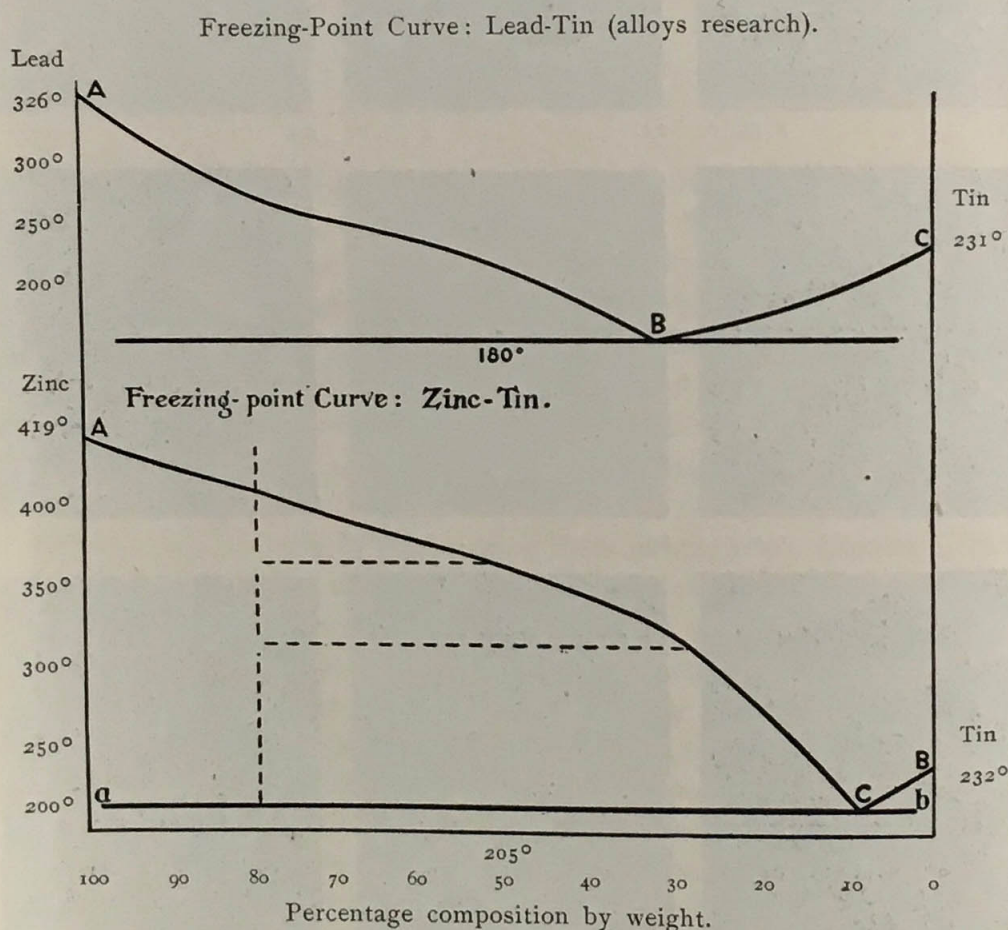


Fig. 33.

giving an alloy, Pb 63 per cent, Sn 31 per cent, Cu 5 per cent, in which three constituents can be seen. The long, rough crystals of a compound of copper and tin have crystallized out first of all; then the excess of lead has solidified as grains and dendrites, and last of all, the eutectic has solidified.

## LEAD AND ANTIMONY

The curve of fusibility is composed of two branches which cut at a sharp angle at the eutectic point, 13 per cent Sb at  $228^{\circ}$  C. according to Roland-Gosselin,\* and  $247^{\circ}$  C. according to Stead.† Fig. 34 shows the curve.

Alloys containing 0 to 13 per cent Sb are extremely difficult to polish and etch properly. They are seen to consist of grains and then dendrites of lead in a matrix composed of alternate bright and dark laminæ, Sb and Pb. Fig. 23 shows the 10 per cent Sb alloy, and the large skeleton crystals of lead are seen to have solidified in the eutectic. This eutectic corresponds to the formula  $Pb_4Sb$ , but this does not mean that a compound exists, but that the eutectic happens to have a percentage composition which corresponds to a formula. An exactly similar case is that of Levul's alloy.

Above 13 per cent Sb the slowly cooled alloys show a distinct layer of harder metal at the upper surface. This hard white layer increases in thickness as the total antimony in the alloy is increased, until at about 50 per cent Sb the whole mass appears bright. Under the microscope these layers are seen to consist of more or less well-formed cubes imbedded in the eutectic. Stead has proved that these crystals crystallize out of a homogeneous mother-liquor, but by gravity float to the top. It is not a case of two liquids, as, for example, lead and zinc.

The crystals, on analysis, were found to contain at most 0.2 per cent Pb, showing that the antimony crystallizes out in almost the pure state, or, in other words, that the concentration of the solid solution of lead in antimony is only 2 in 1000, and that above 0.2 per cent lead we get a eutectic. The specific gravity of these cubes of antimony is 6.5, whilst that of the eutectic is 10.5, which explains why the antimony is found on the top of the slowly cooled alloys. On analyzing the lower portion of any of the alloys from 13 to nearly 50 per cent Sb, Stead found the

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\* Gautier: *Bulletin de la Société d'Encouragement*, 1896, Tome I, 5e Série.

† *Journal Society Chem. Industry*, March and June, 1897. *The Metallographist*, Vol. I (1898), p. 179.



composition to be 12.7 to 12.8 per cent Sb, which gives us the exact eutectic point.

When these alloys are cast the cubes of antimony are comparatively small and are evenly distributed throughout the mass, thus allowing the alloys with the lower percentages of antimony to be used as bearing metals. Fig. 24 shows the surface of an ingot containing 75 per cent Sb, 25 per cent Pb. The cubic growth of crystals of antimony resembles closely that of pure bismuth. As in the other illustrations of surface structures, the eutectic on cooling has shrunk and the antimony crystals stand out in relief. Fig. 30 is very similar to the appearance of the bright layer of antimony cubes in slowly cooled alloys containing from 15 to 30 per cent of antimony.

#### TIN AND BISMUTH

The curve consists of two branches meeting at  $143^{\circ}$  C.\* (Rudberg). The eutectic contains about 46 per cent bismuth, 54 per cent tin, and under high powers has a peculiar granular appearance. From tin to the eutectic we find grains and then dendrites of tin crystallizing out in the eutectic. The alloys are similar in appearance to Fig. 23.

Above 46 per cent Bi, irregular white crystals of bismuth make their appearance. In this case, however, their density is greater than that of the eutectic, and consequently they form and sink to the bottom when the alloy is slowly cooled. When cast they appear regularly throughout the mass. The cooling curve, Fig. 34, is based upon Dr. Gautier's figures in the "Fusibility of Metallic Alloys."

#### ZINC AND ALUMINIUM

The two branches which compose the curve meet at about 5 per cent Al. The eutectic melts at  $380^{\circ}$  C., some  $39^{\circ}$  below zinc, according to Heycock and Neville,<sup>†</sup> or  $389^{\circ}$  C., according to Roland-Gosselin. These alloys are extremely difficult to polish.

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\* *Poggendorf's Annalen der Physik und Chemie*, XVIII, 240. *Annales de Chimie et Physique* [2], XLVIII, 353.

<sup>†</sup> *Journal Chem. Soc.*, 1897, p. 383.

They are best etched with nitric acid, towards the zinc end of the series, and with caustic soda at the aluminium end. The alloys consist of an excess of Al or of zinc in a eutectic consisting of laminae of the two metals. Fig. 25 shows the alloy 4 per cent Al, 96 per cent Zn. Grains of zinc appear in the typical eutectic.

Cooling Curves: Antimony-Lead.  
Bismuth-Tin.

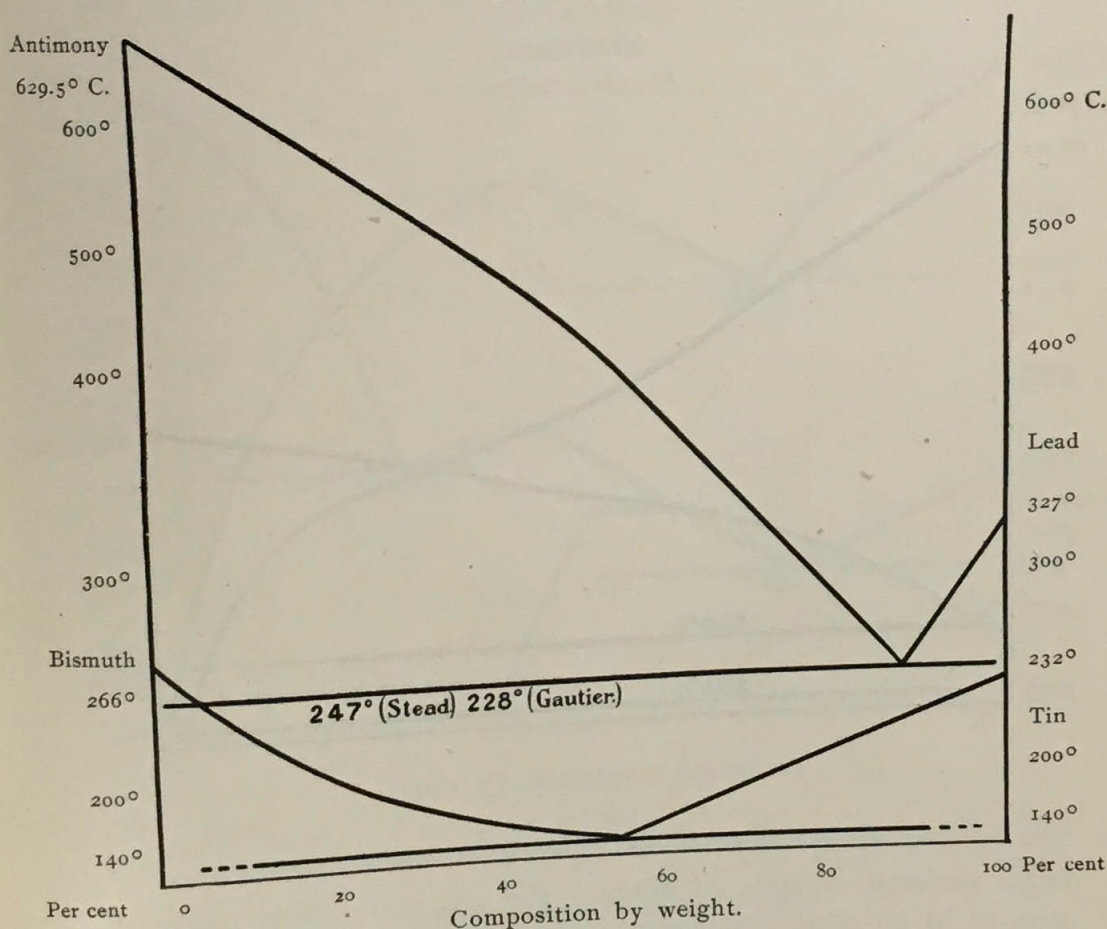


Fig. 34.

### SILVER AND LEAD

The curve consists of two branches, meeting at 2.8 per cent Ag, the eutectic point. This melts at 303° (Heycock and Neville), and the eutectic line extends from almost pure lead to about 96 per cent Ag. Between 0 and 2.8 per cent Ag we find



lead crystallizing out first as rounded grains, then as dendrites in an increasing groundmass consisting of alternate bright and dark laminæ.

Above 3 per cent of Ag, bright white cubes make their appearance, together with well-formed octahedrons. These are silver, but contain some lead in solid solution. As the silver contents are increased, these octahedrons increase, tending to crystallize out along definite directions, and forming in this way

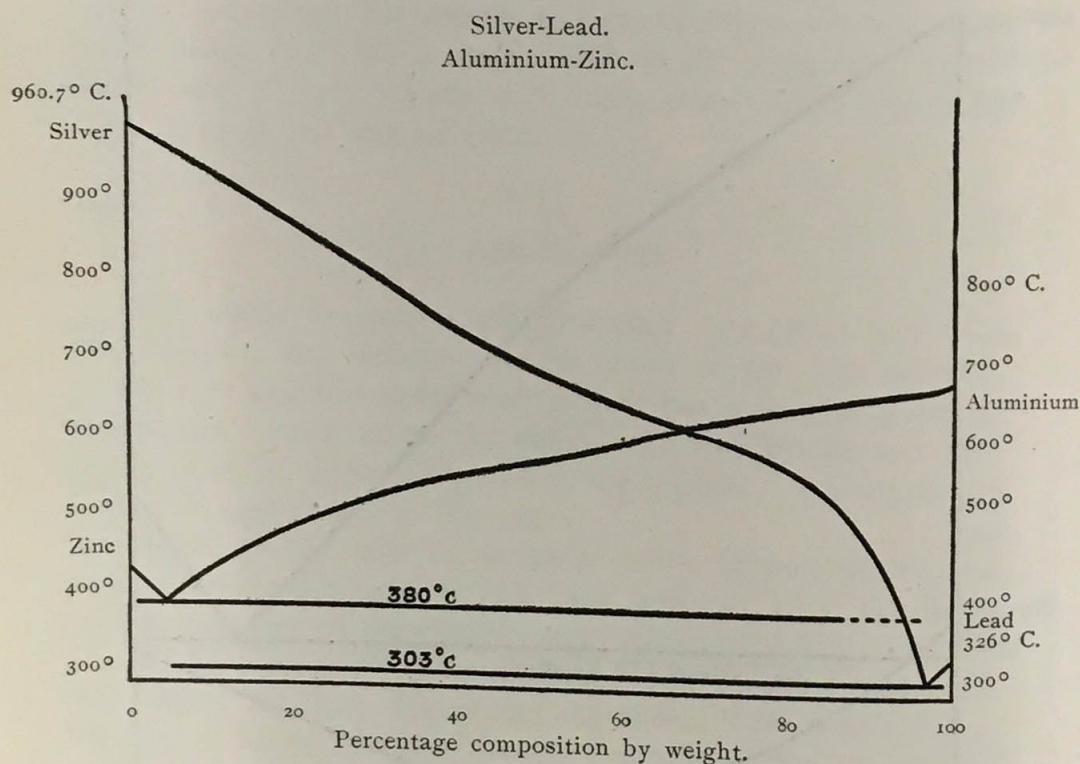


Fig. 35.

large skeleton crystals of silver. Fig. 26 shows this mode of growth in an alloy containing 10 per cent Ag, 90 per cent Pb. The skeleton crystals become more compact, the octahedrons forming them become less perfect, and there is a great amount of interference as the silver is increased. At 50 per cent the silver forms a dense mesh throughout the alloy. This increases in thickness and the eutectic diminishes till at 96 per cent Ag the whole mass appears to be made up of silver grains and dendrites. These alloys are best etched by prolonged action of acetic acid.

## ANTIMONY AND COPPER

The curve of fusibility, as determined by Le Chatelier, consists of three branches, which cross at the two eutectic points about 25 and 71 per cent Cu. The summit of the middle branch

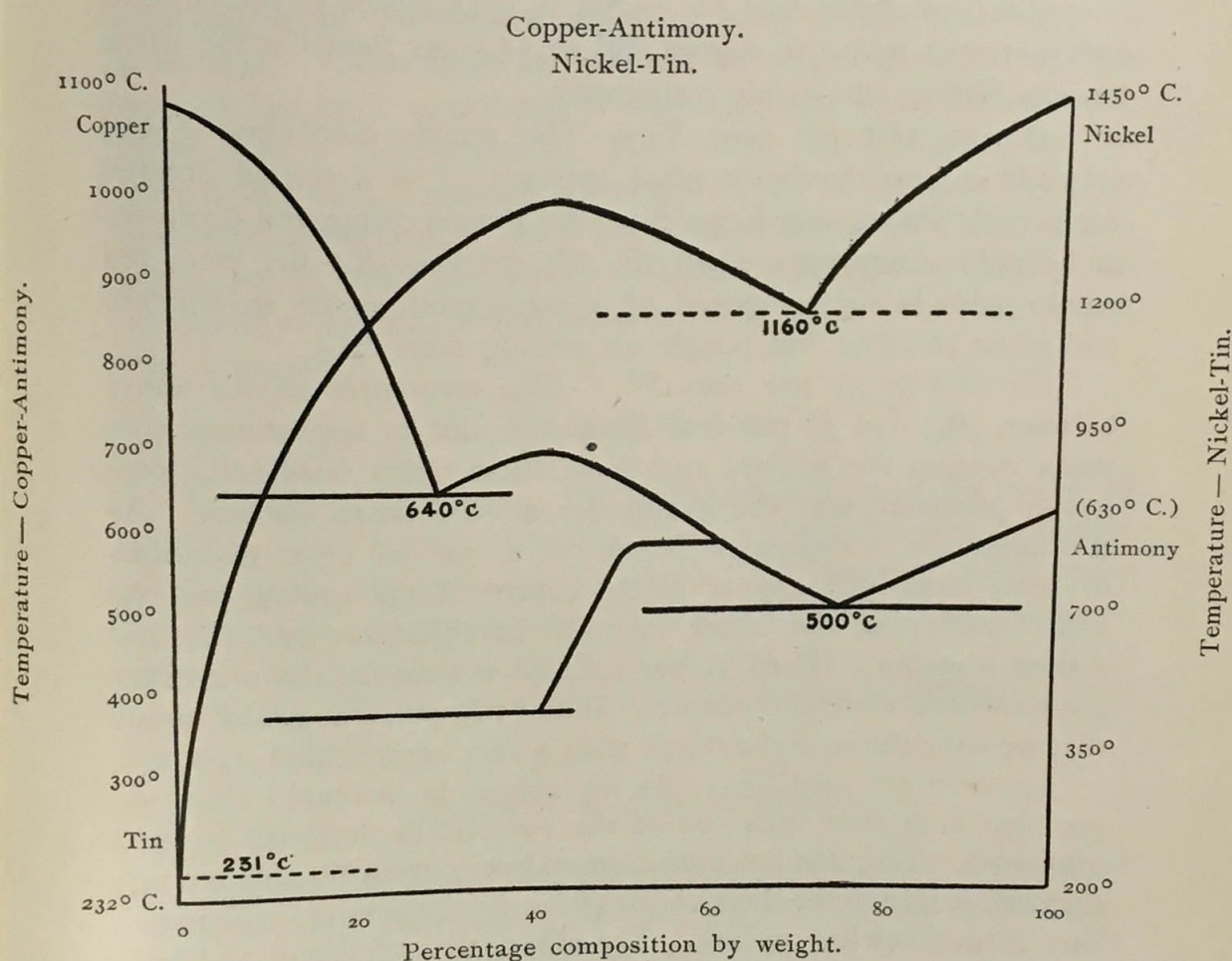


Fig. 36.

occurs at about 60 per cent Cu. According to Stansfield, the two eutectic points occur at 25 and about 69 per cent Cu, and the summit at about 57 per cent Cu. Fig. 36 shows Dr. Stansfield's curve. The lower branches have as yet not been accounted for; but quenching the alloys involved, after complete solidification, but above the lower breaks, revealed new structures and



this seems to point to a rearrangement in the solid similar to that in the copper-tin series.

With regard to the microstructure of the series, Charpy and Stead differ with respect to the alloys near the intermediate summit. Stead's work is summarized as follows:

*100 to 75.8 per cent Sb.* — Antimony crystallizes in a eutectic of 24.2 per cent Cu.

*75.8 to 48.5 per cent Sb.* —  $\text{SbCu}_2$  crystallizes in the eutectic, and increases with the copper, till at 51.5 per cent Cu the whole mass is  $\text{SbCu}_2$ , the purple compound.

*48.5 to 38.5 per cent Sb.* — The purple compound  $\text{SbCu}_2$  crystallizes imperfectly in what appears to be a second definite compound,  $\text{SbCu}_3$ , which is white; the purple compound decreases and finally disappears when the Sb reaches 38.5 per cent, the whole mass being composed of a compound, white in fracture and when polished, but purple on etching with HCl.

*Sb 38.5 to 31 per cent Sb.* — The structures of the alloys between 38.5 and 36 per cent Sb are similar in appearance; thin veins envelop the grains, and from these veins needle-like processes penetrate into the grains for a very short distance. As the antimony is decreased below 36 to 31 per cent, microliths of some compound appear in the centre of the grains, and the veins enveloping the latter increase in thickness and in their copper contents. When 31 per cent Sb is present, the microliths are present in increased quantity, and at this point it would appear that we have the second eutectic with a very complicated structure.

*31 to 0 per cent Sb.* — As the copper is increased above 69 per cent it at first falls out of the eutectic in detached isolated globulites. They are not pure copper, but contain Sb and arrange themselves in definite lines and angles. As the copper is increased, they form dendritic crystals. The microliths are absent and have probably been absorbed by the yellow-colored dendrites at the moment of solidification. The color of the dendrites passes from yellow to red as the Sb is further reduced, and finally the last 0.1 or 0.2 per cent are left behind at the borders of, but not separated from, the grains of copper, where it probably exists as antimonide of copper in solid solution. (Stead: *Journal Soc. Chem. Industry*, Dec. 31, 1898.)

Charpy's conclusions are as follows:

In the case of alloys containing less than 25 per cent copper,



pure crystals of antimony are separated when solidification begins, increasing gradually in size as the temperature decreases, the portion remaining liquid, therefore, gradually becomes richer in copper until the composition of the eutectic alloy is reached; it then solidifies at a constant temperature, through a simultaneous crystallization of Sb and  $\text{SbCu}_2$ . In the case of alloys containing from 25 per cent to 60 per cent Cu, a similar phenomenon occurs, only it is the definite compound  $\text{SbCu}_2$  which separates from the molten mass as soon as the freezing-point is reached. When from 60 per cent to 70 per cent of copper is reached, the same compound is separated, but is in this case surrounded by a second eutectic alloy made up of copper and the compound  $\text{SbCu}_2$ . Finally, when more than 70 per cent of copper is present, a portion of the latter is first deposited when solidification set in, until the portion remaining liquid has reached the composition of the second eutectic alloy (*The Metallographist*, Vol. I, p. 100).

#### TIN AND NICKEL

According to Charpy, the microstructure and the curve of fusibility seem to show that they have a constitution very similar to that of the copper-antimony alloys. The eutectic points occur at 2 per cent and 70 per cent Ni, whilst the summit of the intermediate curve occurs at about 43 per cent Ni.

In Fig. 36 the curve according to Gautier is shown.

#### ALUMINIUM AND ANTIMONY

Gautier points out that the curve of fusibility of these alloys is remarkable, since nearly all its points correspond to temperatures higher than those of the fusion of the two metals. He says that the curve indicates the formation of a compound  $\text{SbAl}$ , whose fusion-point is slightly lower than that of copper. Dr. Mathew's curve is shown in Fig. 12 of his paper.

On a microscopic examination these alloys fall into two groups:

(1) 0 to 81.5 per cent antimony, in which increasing amounts of the compound  $\text{SbAl}$  crystallize out, first as short rod-like crystals, then as more or less irregular bars and plates with much



parallel growth. The groundmass is aluminium or aluminium containing some SbAl in solid solution. The groundmass disappears between 75 and 80 per cent Sb and the alloy appears homogeneous.

(2) 81.5 to 100 per cent antimony, in which we pass from the compound through a series of alloys composed of crystals of SbAl in an increasing groundmass till we reach pure antimony.

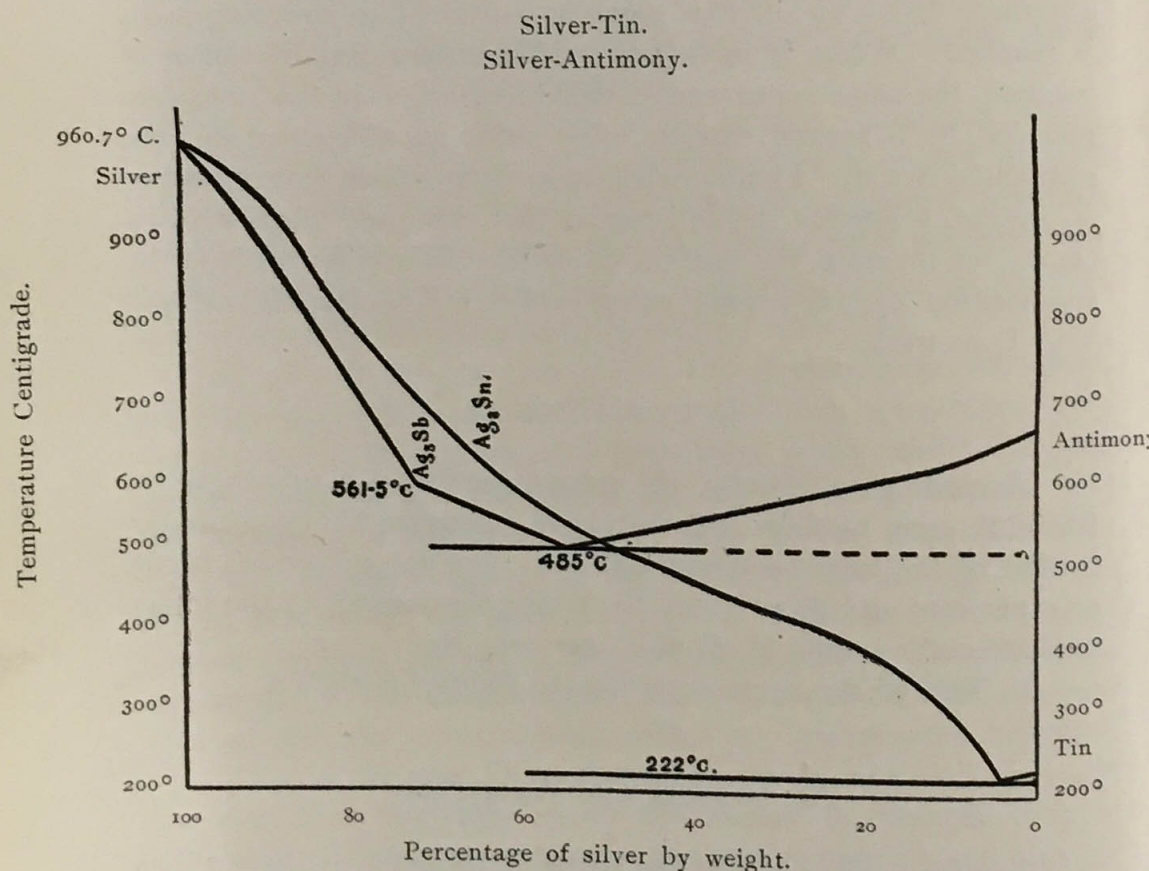


Fig. 37.

The groundmass could not be resolved into two components, and probably consists of antimony containing some of the compound in solid solution.

The series is remarkable in many ways; for when the antimony reaches about 60 per cent the alloys soon become rotten and rapidly disintegrate into a fine black powder. This is due to oxidation, according to Gautier; for if the alloy be weighed before and after disintegration it will be found to have gained in weight.

If the fresh alloy be sealed up *in vacuo*, no change takes place. Again, when the antimony in the alloy is between 50 and 80 per cent a great expansion takes place during solidification and part of the liquid residue is squeezed out and solidifies as a button on top of the alloy. That it takes place during the solidification of the crystals of  $\text{SbAl}$  is shown by the fact that the button contains a large percentage of the compound as well formed crystals. Lastly, it is seen that the curve consists of a rise from the aluminium end to the alloy containing 33 per cent Sb. This is a summit, and the curve falls to 37 per cent Sb, after which it rises again to 81.5 per cent Sb. The reason for this has not yet been explained, for under the microscope the alloys between 33 and 40 per cent Sb present the same characteristics. A similar thing occurs in the alloys of tin and aluminium.

#### SILVER AND TIN

The curve, like that of the silver-lead series, consists of two branches, the one curved, meeting at the eutectic point 3.5 per cent Ag. The eutectic melts at  $222^{\circ}\text{C}$ . and extends from almost pure tin to 65 per cent Ag, where it disappears. According to Charpy, we have a compound  $\text{Ag}_2\text{Sn}$  at 65 per cent Ag, and thus the series can be divided into—

(1) Alloys which are isomorphous mixtures of Ag and  $\text{Ag}_2\text{Sn}$  — between 100 and 65 per cent Ag.

(2) Alloys of the compound  $\text{Ag}_2\text{Sn}$  and Sn, which are quite normal.

Between 0 and 3.5 per cent Ag we find the excess of tin crystallizing out as grains in the eutectic. Above 3.5 per cent Ag we find long, slender needles, probably of the compound  $\text{Ag}_2\text{Sn}$  crystallizing out. The number and size of these hard, bright crystals increase with the total silver in the alloy. They assume curious tree-like shapes, finally becoming united and forming dendrites. At about 50 per cent they occur as rounded grains in close contact. The eutectic diminishes, and at 65 per cent Ag we find the alloy homogeneous. This alloy may either be a definite compound or it may be a solid solution of tin in silver. From this point onwards to pure silver the alloys are isomorphous with silver. Fig. 29 shows an alloy of the first



group containing 15 per cent Ag, 85 per cent Sn, and contains the curiously grouped crystals of the compound set in the eutectic. The curve, Fig. 37, is based on the freezing-point curve of Heycock and Neville.

#### ANTIMONY AND SILVER

In Fig. 37 we have the cooling curve based on the figures of Heycock and Neville. It is composed of two branches: the one from pure antimony to 55 per cent Ag, the eutectic point, being normal; the other from silver, having a decided angle at 72 per cent Ag, corresponding to the formula  $\text{Ag}_3\text{Sb}$ . The eutectic melts at  $485^\circ \text{C}$ ., whilst the 72 per cent Ag alloy melts at  $561.5^\circ \text{C}$ .

According to Charpy, between 72 per cent Ag and 0 per cent Ag we have excess of either  $\text{Ag}_3\text{Sb}$  or Sb crystallizing out in the eutectic, in the same manner as all the alloys of Group I; between  $\text{Ag}_3\text{Sb}$  and pure silver we have a series of isomorphous mixtures of the definite compound with silver. It may be, however, that here we are dealing with a case similar to the antimony end of SbSn series, and that we have dendrites of silver crystallizing out in the compound  $\text{Ag}_3\text{Sb}$ . This point will be settled when a complete cooling curve has been taken of this end of the series.

#### TIN AND ANTIMONY

Between 0 and 7.5 per cent of antimony the alloys crystallize out in the same forms as pure tin; in other words, the tin will retain some 7.5 per cent of Sb in solid solution, probably in the form of the compound SbSn. When the antimony is increased above 7.5 per cent, some very hard, bright cubes are found at the surface of the alloy when slowly cooled, but when cast the cubes are found scattered throughout the alloy. The bright layer of cubes increases in thickness with the antimony in the alloy, till at about 30 per cent Sb it reaches the base of the alloy, and the groundmass is seen occupying the interstitial spaces. At about 40 per cent the form of the cubes begins to change, and at 45 per cent Sb the alloy consists of squat, thick bars or plates crossing at all angles, the spaces between being still occupied by a groundmass similar to that found in the lower percentage alloys.



At about 52 per cent Sb, a new constituent, probably antimony, is found making up the core of the bars or plates, but there still remain traces of the groundmass, which finally disappears at about 55 per cent Sb. The cores of the bars continue to increase and develop into the usual crystals of antimony and, at about 95 per cent Sb, become continuous. In the joints between them traces of a matrix are seen, which finally disappear as we approach pure antimony.

Fig. 30 shows a vertical section through the centre of a slowly cooled alloy containing 20 per cent Sb, 80 per cent Sn. The bright cubes are seen set in the softer matrix. Stead found that the common freezing point of this matrix between 7.5 per cent and 50 per cent Sb was  $256^{\circ}$  C., which is remarkable since it is higher than that of pure tin by  $25^{\circ}$  C. On analyzing the cubes isolated from a 25 per cent Sb alloy, he found their composition to be approximately SbSn. He says that a homogeneous mass corresponding to SbSn cannot be obtained, for on melting the metals in that proportion the resulting alloy consisted of the peculiar plates and the dark matrix; that the crystals up to 30 per cent Sb are SbSn, but in the neighborhood of 40 per cent Sb the forms begin to change and the contents of antimony increase. This is contrary to Behrens,\* who isolated from the 10 per cent alloy cubes with the formula  $\text{SbSn}_2$ , and from the 64 per cent Sb alloy a residue of the formula SbSn.

According to Stead, the specific gravity of the cubes is 6.96, which is lighter than that of the groundmass, and thus they float to the top of the slowly cooled alloys, just as the antimony in the lead-antimony alloys. Fig. 31 shows the surface structure of the cast 25 per cent Sb alloy, in which the cubes are quite distinct. In Fig. 32 we have the 70 per cent Sb alloy, showing the crystals of antimony set in a matrix, which is probably SbSn, containing much antimony in solid solution. Stead puts forward the alternate view that Sb and SbSn may form isomorphous compounds with each other, and when the antimony reaches a certain point it crystallizes out in a separate state.

The alloys of tin and phosphorus and of tin and arsenic probably belong to the same group as tin and antimony.

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\* *The Metallographist*, Vol. III, p. 11.



## TIN AND PHOSPHORUS. TIN AND ARSENIC \*

When phosphorus is added to tin, a hard constituent, consisting of brilliant white plate, similar to graphite, is formed. Stead has studied the series from 0.04 per cent to 5 per cent

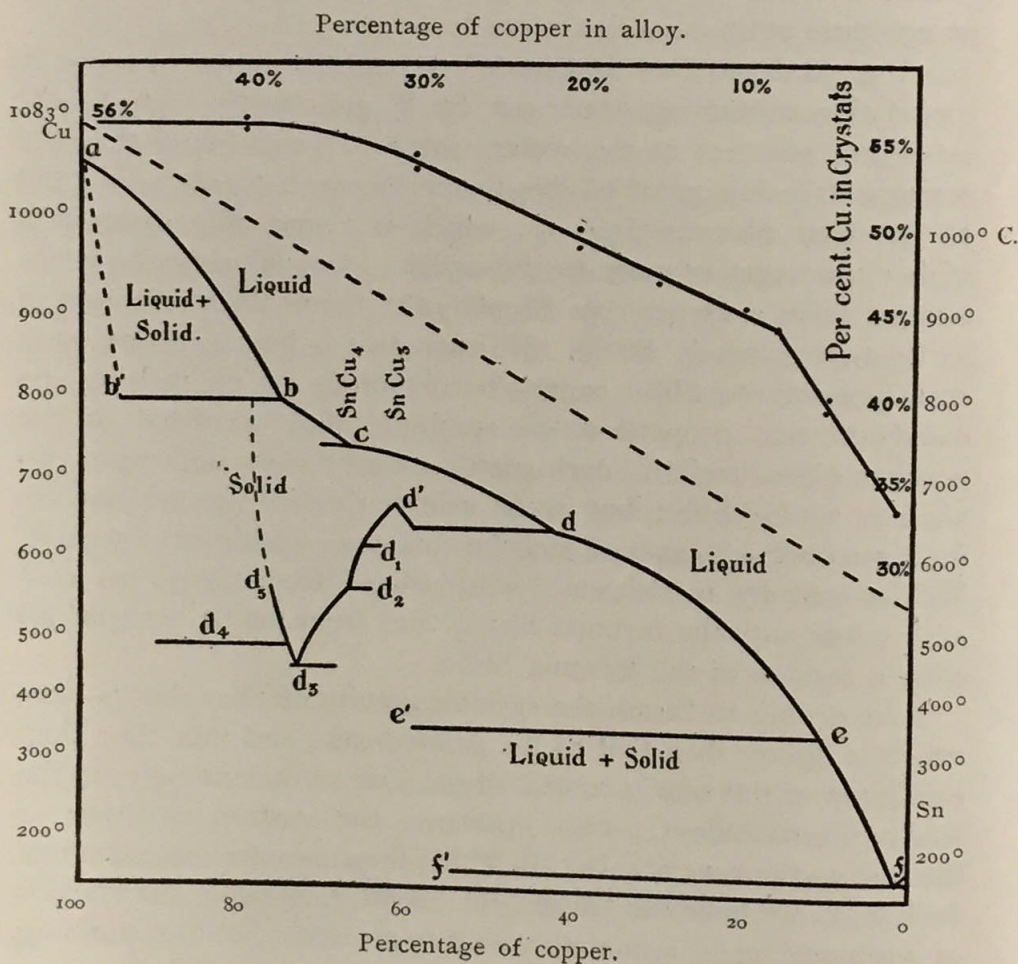


Fig. 38.

phosphorus. The structure of these alloys can be developed by polishing alone, for the phosphide formed stands out in relief from the soft matrix of tin between. On etching with dilute acid this groundmass of tin (probably containing some phosphorus

\* Stead: *Journal Society Chemical Industry*, March, 1897. *The Metallographist*, Vol. I (1898), p. 179.

in solid solution, for its melting point is  $4^{\circ}$  C. above that of pure tin), turns black, and the hard white crystals of phosphide stand out in strong contrast. When analyzed the bright white plates proved to have the composition  $\text{Sn}_3\text{P}_2$ . When the alloy is slowly cooled the phosphide invariably commences to grow at the outer portion of the alloy and travels in straight lines toward the centre. The plates have a decidedly hexagonal form. Fig. 28 shows an alloy containing 2 per cent phosphorus; it has been cast, and so the phosphide appears as comparatively small thin plates throughout the groundmass.

When arsenic is added to tin, thick rough plates are formed, having the composition  $\text{Sn}_3\text{As}_2$ . The groundmass consists of tin, probably containing some arsenic in solid solution, because, as in the case of phosphor-tin, the melting point has been raised some  $4^{\circ}$  C. Stead was able to introduce as much as 43 per cent As into tin. Fig. 27 shows a slowly cooled alloy, in which the thick rough plates of arsenide run in all directions, with a tendency towards parallel growth. The alloy contains 20 per cent As.

#### COPPER AND TIN

The microstructure of the copper-tin alloys has been studied by Behrens, Charpy, Stead, Heycock and Neville. The earlier explanations were based on Le Chatelier's curve of fusibility, which consists of three branches forming by their intersections two points corresponding to alloys with 3 and 72 per cent of copper. These two points correspond to the two eutectic alloys of the series. The existence of the compound  $\text{SnCu}_3$  (61.7 per cent Cu) has been proved, for at this point we find discontinuities in the variations of many properties, such as the electromotive force of dissolution (Laurie); the electric conductivity (Mathiesen); the specific gravity (Riche). Hence it was thought that the series consisted of two simple sets of alloys, viz.: the alloys of copper and  $\text{SnCu}_3$  and the alloys of  $\text{SnCu}_3$  and tin, and was similar in structural variation to the alloys of copper and antimony.

This view of the constitution of this series of alloys does not explain the complete freezng-point curve published in the fourth report of the Alloys Research Committee, Institution of



Mechanical Engineers, shown in Fig. 38. It was not until recently that an explanation of the branches *b*, *d* and *e* was offered.

*0 to 1 per cent Copper.* — When 1 per cent of copper is present the first eutectic alloy is obtained; that is, the one with the lowest freezing-point. Between pure tin on the one hand and this alloy containing 1 per cent Cu on the other, tin is found crystallizing, first in grains, then in dendrites in the eutectic. The cooling curves of all the intermediate alloys show two breaks.

*1 to 8 per cent Copper.* — When the percentage of copper is increased above that of the eutectic alloy, thin, bright, hollow crystals are seen. In section they are horseshoe-shaped, and at first occur isolated; then they tend to form groups which appear in section as three- and six-rayed stars. Their composition varies also, increasing in copper from 34 to 44 per cent.

*8 to 40 per cent Copper.* — A third constituent is seen when the copper exceeds 8 per cent. We have the eutectic or ground-mass enclosing the bright porphyritic crystals characteristic of the 1 to 8 per cent alloys, but these bright crystals are seen in places to have grown on and around a different kind of crystal. It is not a case of one crystal varying in composition from the centre to the faces, for a sharp line of junction can be seen between the two constituents. On oxidation this new constituent becomes very dark, and is easily distinguished from the other two constituents of the alloy. As the percentage of copper is increased, the more easily oxidized crystals increase in number and size; whereas the bright crystals begin to decrease together with the eutectic. It would, therefore, appear that in alloys containing more than 8 per cent of copper, the first constituent to crystallize out is the central, easily oxidized crystals. This causes the first rest in the cooling curve *d e*. Then the bright crystals solidify, causing the second halt on the horizontal branch *e' e*. Lastly the eutectic solidifies and the third halt is reached on branch *f' f*. As branch *e' e* is horizontal, it would seem that the bright crystals have a definite composition when above 8 per cent Cu is present in the alloy, but when branch *e' e* joins the outer curve at *e* and falls to *f*, these crystals no longer have a definite composition, but their percentage of copper falls with the temperature of solidification from  $\text{Sn}_2\text{Cu}_3$  to  $\text{SnCu}$ .

The upper curve in Fig. 38 shows the relation between the percentage of copper in the alloy to the percentage of copper in



the crystals which have been isolated. A break occurs at 8 per cent copper, at the introduction of the third constituent to the alloys. These results agree very closely with those of Stead.\*

As the copper approaches 40 per cent, the central plate-like crystals are grouped together in parallel bunches, until at 40 per cent Cu they are very thick and cover more than half the field. In the eutectic between them the small, bright, hollow crystals are seen.

*41 to 61.7 per cent Copper.*—The difference between the alloy containing 40 per cent and that containing 41 per cent Cu is very marked. The crystals in the latter are small and lath-shaped, arranged more or less in groups and are separated from each other by eutectic. They are composite as before, but the white constituent surrounds the dark as an envelope of uniform thickness, not as a rough incrustation. No single prismatic crystals of the white constituent have been seen in the eutectic. With each addition of copper the groups of crystals become more and more compact and the amount of eutectic diminishes until at 56 per cent Cu it disappears altogether.\* Therefore branch  $f f'$  of cooling curve ends at 56 per cent.

The bright constituent of the crystals grows smaller and smaller; at 56 per cent Cu it takes the place of the eutectic and forms the groundmass, whilst at about 61 per cent Cu it disappears and we have a homogeneous mass of  $\text{SnCu}_3$ . Hence branch  $e e'$  ends at 60 per cent at  $e'$ .

Seeing that these alloys up to 56 per cent Cu show four breaks in their cooling curves, one would naturally expect to find four different constituents in each. Only three, however, can be distinguished. Quenching below the first and second breaks gives a difference in structure only. As in the alloys containing 61.7 per cent Cu and onwards, branch  $d$  of the freezing-point curve corresponds to a rearrangement in the solid, and as the difference between the 40 per cent alloy and those of a higher copper contents is one of structure only, we may assume that the second retardation in the cooling curve  $dd'$  is one of rearrangement also.

*61.7 to 68.2 per cent Copper.  $\text{SnCu}_3$  to  $\text{SnCu}_4$ .*—The changes which take place between these two points can only be observed

\* *Loc. cit.*



when the alloys are very slowly cooled. The alloys set as a whole at the first break on  $c d$ , and tend to rearrange themselves subsequently in the solid. Near  $\text{SnCu}_3$  we see the dark grains of this compound surrounded by an envelope of a bright material, probably  $\text{SnCu}_4$ , whose formation is indicated by  $d_1$  in the cooling curves. Each addition of copper brings in more and more of the bright constituent. Above 65 per cent Cu we find, in places, a structure like that of a eutectic which accounts for the horizontal branch  $d_2$  of the curve. At 68.2 per cent Cu we have a homogeneous alloy, very brittle, taking a beautiful polish, and this very probably is a definite compound,  $\text{SnCu}_4$ . When the alloys of this group are quenched at definite temperatures, very many curious and beautiful structures are met with. For example, if the 66 per cent Cu alloy be quenched below the first break, say at  $720^\circ \text{C}$ ., then a clear cell-like structure is seen. If it is quenched between the first and second break, say at  $650^\circ \text{C}$ ., then the structure consists of a network of parallel straight lines, alternately light and dark. Each large area has two or more sets of lines, which are differently orientated for different areas. It may be called the "Schiller" structure of these alloys. If the alloy be quenched below the second break, but above  $d_2$ , say at  $600^\circ \text{C}$ ., the structure is almost the same as that met with in the slowly cooled alloy, except that no sign of any eutectic can be seen. If the 68.2 per cent alloy be quenched just after the first break, its structure is that of dark rounded grains passing imperceptibly into a light-colored groundmass, clearly showing that the alloy is in a metamorphic state. Thus it seems quite certain that in this group the alloys rearrange themselves in the solid.

*68.2 to 75 per cent Copper.* — In this group we pass from the homogeneous  $\text{SnCu}_4$ , consisting of irregular and elongated grains, to the so-called second eutectic at 74 to 75 per cent Cu. Between these two alloys we find the compound  $\text{SnCu}_4$  crystallizing in the eutectic. Near 68.2 per cent Cu the alloys are composed of polygonal grains, with the boundaries of bright  $\text{SnCu}_4$ . Their central parts are composed of dendrites and rosettes of  $\text{SnCu}_4$  set in the eutectic. Near 74 per cent Cu the alloys are composed of attenuated rosettes and grains of  $\text{SnCu}_4$  set in a comparatively large amount of eutectic. Thus the upper part of branch  $d$ , between  $d_2$  and  $d_3$ , marks the point at which these alloys rearranged themselves into grains isomorphous with  $\text{SnCu}_4$ ,



whilst the horizontal branch  $d_3$  marks the formation of the eutectic, both changes having taken place in the solid. At  $c$  there occurs a small horizontal branch, the meaning of which is not quite certain. A microsection of an alloy quenched between this and the outer curve shows dark rounded grains set in a light ground-mass. Heycock and Neville say that the outer curve  $bc$  indicates the formation of skeletons rich in copper; but when the alloys are quenched below the horizontal branch  $c$ , then we have uniform solid solutions. It is to be noted that on the surfaces of alloys containing 70 per cent or more copper, a network of dendrites or skeleton crystals resembling those on the surface of a pure metal is to be seen. It was soon noticed that the internal structure of the alloys from 70 to 75 per cent Cu showed no trace of these dendrites, and so the surfaces of several were rubbed down, polished and etched so as to lay bare their internal structure. In each case it was the same as that of the centre of the alloy, which shows that these dendrites have split up and rearranged themselves after solidification, and all that remains of them is this surface structure.

*75 to 100 per cent Copper.* — Above 75 per cent Cu two new constituents make their appearance and the alloy assumes a yellow tint and begins to lose its brittleness. In section we find yellow grains surrounded by a bright white border, set in the second grains surrounded by a bright white border, set in the second eutectic, in which small white grains also occur. Now this eutectic at 76 per cent Cu is much larger in character than the 74 per cent alloy, and this may account for the fact that the eutectic break rises some  $30^\circ$  C. as it passes from 74 to 75 per cent Cu.

As the total copper is increased the yellow grains increase, forming dendrites and skeleton crystals, the white borders and grains merge together, and the eutectic decreases till at about 90 per cent Cu it disappears. The yellow grains of copper become darker and darker (contain less and less tin in solid solution) till they reach copper color. The light borders diminish and disappear about 95 per cent, leaving copper dendrites alone. These dendrites vary in composition from centre to outside, and so the centre etches a darker color. These dendrites darken with the increase of copper till about 100 per cent is reached, when we have the characteristic structure and color of pure copper.

Quenching these alloys at different temperatures proves that copper grains and dendrites begin to crystallize out as soon as



an alloy has reached the temperature of the outer curve  $ab$ ; that these continue to grow till the temperature falls to  $b'b$ , when the whole mass becomes solid; at this point we have crystals of copper in a homogeneous groundmass. Above the curve  $ab$  the alloy is liquid, below  $b'b$  the alloy is solid, whilst between  $ab$  and  $b'b$  we have a mixture of solid and liquid. The composition of the copper dendrites crystallizing out of any particular alloy would be given by the composition line  $ab'$ .

Below  $b'b$  the groundmass consists of a solid solution much richer in tin than the grains of copper in it. It is probable that these grains of copper continue to grow in the solid until at about  $500^{\circ}$  C the groundmass has arrived at the composition 75 per cent Cu, when it splits up into a eutectic composed of laminae of  $\text{SnCu}_4$  and Cu, containing a considerable amount of Sn in solid solution. This change in the solid is marked by branch  $d_4$ .

The meaning of branch  $d_5$  is obscure. It may indicate the formation of the bright white grains seen in the 76 and 77 per cent alloys.

In their recent papers before the Royal Society of London and elsewhere, Heycock and Neville\* have clearly proved that the great changes which take place when a bronze reaches the temperature of the curve  $d' \dots d_4$  are entirely in the solid. They go further than this by determining the various substances or phases which exist in the various alloys, either chilled or slowly cooled. Their modification of the cooling-curve diagram by the addition of a curve indicating the position of the *end* of solidification of each alloy and their explanation of this new diagram, go far in clearing up many of the numerous difficulties met with in the alloys of copper and tin.

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\* Proceedings Royal Society of London, Vol. LXVIII. British Association, Glasgow Meeting, 1901, Chemical Section.

## A NEW FORM OF RECORDING APPARATUS FOR A ROBERTS-AUSTIN ELECTRICAL PYROMETER

By ERNEST A. LEWIS

THE usual form of recording apparatus for this pyrometer, which is used in works for taking continuous records of furnace temperatures, is of no use for taking cooling curves of small quantities of metals. It consists of a drum worked by internal clockwork carrying a sheet of bromide paper, which turns round once in 6, 12, or 24 hours.

The author has designed the following additional apparatus to the pyrometer in the laboratory of Muntz's Metal Co., Ltd., so that the same pyrometer can be used to take furnace temperatures and also cooling curves of small weights of molten metals solidifying in a crucible.

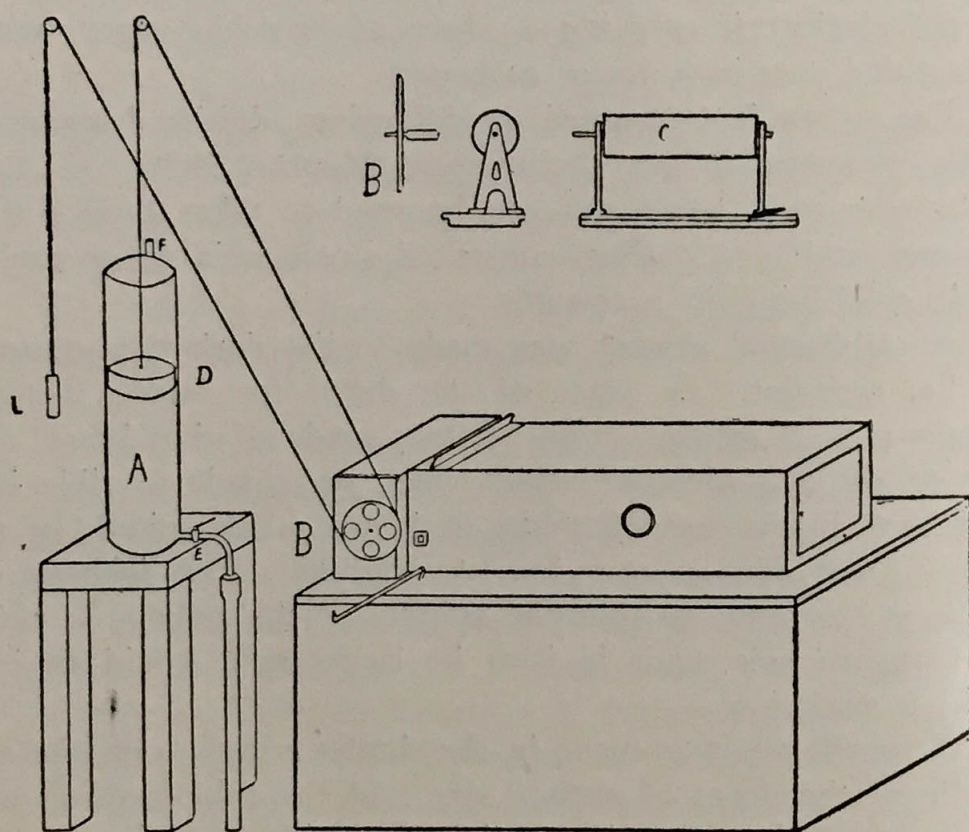
An additional shutter was made to the dark box containing the recorder. In place of the drum for taking furnace temperatures, a smaller drum, C, was made of thin brass, and screwed on to a piece of wood which was made to slide into the groove in the bottom of the dark box. The spindle of the drum C was filed square for five eighths of an inch of its length at one end, so that the spindle of the pulley B which has a square hole, made in it at its larger end, would fit into it like a clock key.

A round hole was made in the shutter exactly opposite the spindle of the drum of such a size that the spindle of B will just go through and turn round easily, without letting in any light. Over the grooved pulley B runs a cord which passes over two small pulleys screwed into the ceiling; one end of the cord is attached to a float D, which slides easily inside the cylinder A; to the other end of the cord is attached a leaden weight L. The cylinder A in the author's apparatus is made of a copper tube; on the one side is a supply pipe F, which is screwed into the cylinder A at the bottom; this serves to run water into A. The tap E is connected to a waste pipe.

To use the apparatus for taking a cooling curve a whole-plate extra rapid film is put on the drum C, and fastened with a clip similar to the clip on the furnace record drum. The drum is then slid into the groove at the bottom of the dark box, and



the shutter put in. The pulley B is put through the hole in the shutter into the squared end of the spindle of the drum. The drum is turned round until the end of the film is judged to be nearly opposite the slit in the pyrometer case. A little practice enables this to be easily done as the weight of the clip of the drum always causes the clip to be at the bottom, when the drum is put in. The cylinder A is filled with water and the float D



RECORDING APPARATUS.

put in, the cord is then put round the pulley B and over the two small pulleys in the ceiling as shown in the diagram. The pulleys and bearings of the drum must be well oiled, also the hole in the shutter through which the pulley B spindle passes must be oiled, so as to avoid friction.

The door at the end of the pyrometer is opened to obtain a datum line, and the gas jet of the pyrometer is lighted; when the thermo-junction is put into the molten metal the tap E is opened, and the float descends and turns the drum with its film round.



The speed at which it turns is regulated by the rate of outflow of the water. It can be made to turn round once in from five minutes to one hour. The weight of the pulley B causes the drum to turn round absolutely steady. In order to test the sensitiveness of the recorder, the author made an alloy of copper 50 per cent, tin 50 per cent, which is known to have four freezing points at  $680^{\circ}\text{C.}$ ,  $635^{\circ}\text{C.}$ ,  $410^{\circ}\text{C.}$ , and  $225^{\circ}\text{C.}$  Each of these breaks in the cooling curve were distinctly marked on the film.

The following are the principal dimensions of the recorder:

Cylinder A, 2 ft. 8 ins. high  $\times$   $8\frac{1}{2}$  ins. internal diameter.

Float D,  $8\frac{1}{4}$  ins. diameter  $\times$   $2\frac{1}{4}$  ins. thick weighted with lead to 4 lbs.

Balance weight L,  $2\frac{1}{4}$  lbs.

Pulley B, 7 ins. diameter  $\times$   $\frac{1}{4}$  in. thick, groove  $\frac{1}{8}$  in. on edge.

Spindle of B, 3 ins. long  $+$   $\frac{7}{16}$  in. diameter at large end,  $\frac{1}{4}$  in. diameter small end.

Drum,  $9\frac{1}{8}$  ins. long  $\times$   $2\frac{1}{8}$  ins. diameter.

Spindle of drum,  $11\frac{1}{4}$  ins. long  $\times$   $\frac{1}{4}$  in. diameter.

The author has found this recorder to give cooling curves which are of great value in determining the correct temperature at which metals must be rolled. The recorder is simple in construction, and is easy to work. The pyrometer should be kept in a room, the window of which is covered with two thicknesses of ruby fabric; this enables the films to be put in and developed in the pyrometer room.

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## METALLOGRAPHIC NOTES

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**Alloys of Copper and Tin.** — The reports of the Alloys Research Committee of the Institution of Mechanical Engineers are rightly regarded as classical contributions to the modern store of knowledge concerning the constitution and properties of these important materials of construction, and to these we now have the appendix, as it is called, in the form of the paper recently



presented before the Institution by Mr. William Campbell upon the alloys of copper and tin.

These researches were undertaken in order to investigate more fully the freezing-point curve of the copper-tin alloys as published in the fourth report of the Alloys Research Committee, and to note the change of structure due to casting.

Without going too deeply into the technical side of this important subject, the general character of the investigations may be here discussed, and the methods of work described, together with some examination of the practical importance of the results to the engineer and constructor, while the metallurgist and physicist may well study the original paper as an example of painstaking scientific work.

The original curve of fusibility of the copper-tin alloys was determined by Le Chatelier, and was shown to consist of three branches, changes in direction taking place at 3 per cent and at 75 per cent of copper. The later investigations of Sir William Roberts-Austen have added other points to the curve which evidently indicate changes in molecular condition, and it was to investigate the nature and constitution of the alloys at these points that the investigations of Mr. Campbell were undertaken.

By polishing and etching various specimens, their physical constitution may be studied under the microscope, and the object of Mr. Campbell's researches was to prepare the various alloys which showed peculiarities in the cooling curve, and to examine them by the methods of metallography to discover, if possible, the causes of the peculiarities, or at least the structural conditions which accompanied them.

The apparatus used in the researches is fully described in the paper, and consisted of a pyrometer of the Le Chatelier type, the galvanometer reflecting a spot of light upon a scale which could be watched as the cooling took place, and the operation interrupted at any desired temperature. The method of operation consisted in the very careful preparation of the various alloys, their fusion in a double crucible, a smaller one being placed inside of a larger and the space between being filled with asbestos, so as to insure slow cooling. In this way the average time of the fall in temperature from  $1,800^{\circ}$  to  $900^{\circ}$  F. was about half an hour. When the melted alloy was removed from the fire, together with the outer crucible and lid, the



thermo-couple of the pyrometer was pushed through a hole in the lid, into the molten metal. When the spot of light reflected from the pyrometer showed on the scale the desired temperature, the wires of the thermo-couple were withdrawn quickly, and the inner crucible and its contents plunged into cold water.

By this method a great number of specimens of varying content of copper and tin were obtained, each having been slowly cooled to a determined temperature, and then suddenly chilled, so as to secure the structure at that point, and these specimens were then polished and etched and examined under the microscope. A large number of engravings from micro-photographs of the various alloys accompany Mr. Campbell's paper, and these, with his comments, enable the researches to be intelligently followed. While the study of these must be left for the student who desires to follow the work in detail, some of the general conclusions may here be given.

There appears to be no doubt that most important structural changes take place in solid alloys as the result of heat treatment, and while it may be necessary for the microscopical examination to be supplemented by tensile and compression tests, before the results become fully applicable to the engineer and constructor, yet much of practical value is already shown.

In the course of the discussion, Mr. Milton, of Lloyd's, called attention to the value of the cooling curves of the various alloys as revealing the purity of the metals employed. The researches of Roberts-Austen indicate that in the case of pure metals the breaks which appear in the curves are perfectly horizontal, while with impure metals they are curved, or present rounded corners. The explanation is that at the temperature at which the break occurs a certain number of molecules of a definite compound of copper and tin become solidified, and when the temperature falls a few degrees a few others solidify, these being of a composition richer in tin than the first, and forming a layer around them. There are thus formed successive layers of increasing richness in tin, according to the rate of cooling.

**Importance of Heat Treatment.**—It is frequently inferred that the quality of steel depends primarily upon its chemical composition, or at least, that the conditions of manufacture other than those related to its composition, are easily met, being



in the majority of cases satisfactorily fulfilled by the manufacturers. Long ago, however, the experiments of Brinell and Tschernoff, and, more recently, those of Sauveur and Howe, have shown the great importance of heat treatment. Outside of large iron and steel works, nevertheless, these new laws are but little known. The consumers remain ill informed on this subject, and do not always require from the steel works what the latter are now able to give them without much extra effort, provided they are asked with some insistence. More frequently still, consumers spoil, through defective treatment, metals of good quality supplied to them by the makers.

The experiments conducted during the last few years to ascertain the brittleness of metals, by breaking nicked bars of these metals under a falling weight, have proved an important source of information to the consumer. These tests reveal that some steel bars, having an excellent composition, break so readily under shock as to indicate a discouraging degree of brittleness, while when subjected to a tensile test the results are very satisfactory. The failure of steel implements when in service, and which remain so often unaccounted for, being generally attributed to some local defects, are a direct result of this brittleness. It is indeed surprising that such accidents are not more frequent, and in view of these facts it is of much importance to make known, as far as possible, the knowledge which we have of the proper heat treatment of steel. — *H. Le Chatelier, Bulletin Société d'Encouragement, July, 1902.*

The use of the microscope for the purpose of ascertaining the proper heats for hardening and annealing has done much to educate the steel user in proper treatment. To-day there are practical men in almost every large establishment where tools are made, who can offer a logical explanation as to why steel hardens. — *Extract from the Development of Tool Steels, by E. L. French, American Association for the Advancement of Science, Pittsburg Meeting, July, 1902.*

**Metallography at the Royal Society.** — Progressive metallurgists must have noticed with pleasure the comparatively large number of exhibits at the recent *soirée* of the Royal Society dealing with the science and art of the micro-structure of metals.



The art of metallography was represented by a neat tilting stage, by the motions of which the surfaces of objects under microscopic observation can be adjusted at right angles to the optic axis, besides being moved about in the ordinary way, and a compact, yet fully equipped, optical bench for metallurgical work — both exhibited by Professor W. K. Huntington. Under the former heading came Mr. T. Andrews' photomicrographs, showing the crystalline structure of platinum and of large steel ingots; and Mr. J. E. Stead's beautiful exhibit of specimens of phosphoretic iron, whose constituents were brought into relief by means of the method of heat-tinted oxidation films which in Mr. Stead's hands has lately yielded such valuable results (see *Electro-Chemist and Metallurgist*, March 1902, p. 47). Interest, however, chiefly centred about Mr. George Beilby's remarkable exhibit of a hitherto unobserved film structure which is to be seen on the surfaces of polished metals and other plastic solids — such as graphite and lead sulphide — when they are viewed under the microscope by powerful oblique illumination at magnifications of from 500 to 1,000 diameters. This covering film, which shows the characteristic colour of the metal (for example, in the case of steel it is blue), is evidently formed by the welding together of minute reflecting surfaces, if one may judge from its spicular appearance. These spicules are specially well seen in surfaces which have been frosted by the action of heat and chemical reagents, but it is remarkable that they should be discernible even on the polished surface. — *The Electro-Chemist and Metallurgist*, May, 1902.

**Metallography in Court.** — An important law suit, Jackson *vs.* Mumford, has just been tried at the court of King's Bench, London, respecting the cause of the fracture of a connecting rod during the trials of H. M. S. *Bullfinch*, about three years ago, whereby eight men were killed. The case largely depended on the quality of the steel, and Professor Arnold gave expert evidence on the microstructure for the plaintiffs, while Mr. Stead appeared for the defendants. We understand that the judgment was reversed.

**Recent Converts to Metallography.** — The Pennsylvania Railroad Co. have installed a very complete metallographic outfit



in their laboratory at Altoona, Pa. The Polytechnic Institute of St. Petersburg and the School of Mines at Ekaterisnoslav, Russia, have also added to their equipment the necessary apparatus for metallographic work.

**Recent Publications.** — *Metallography*, by Arthur H. Hiorns. Macmillan & Co. There are probably few students of metallography who have not wished for the publication of a treatise on the subject, and we believe that most of our readers will be interested to hear that such a book has just appeared. We fear, however, that after a perusal of the book they will be somewhat disappointed, for it contains unmistakable evidence of having been hastily written, and of only an imperfect and fragmentary knowledge of the subject on the part of its author. The treatment is decidedly loose and unscientific, and many of the photomicrographs reproduced are of an inferior character.

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[The names of authors are printed in SMALL CAPITALS and the titles of articles in *italics*]

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